



# Characterization, origin and aggregation behavior of colloids in eutrophic shallow lake



Huacheng Xu <sup>a,\*</sup>, Mengwen Xu <sup>b</sup>, Yani Li <sup>a,c</sup>, Xin Liu <sup>b</sup>, Laodong Guo <sup>d</sup>, Helong Jiang <sup>a</sup>

<sup>a</sup> State Key Laboratory of Lake Science and Environment, Nanjing Institute of Geography and Limnology, Chinese Academy of Sciences, Nanjing 210008, China

<sup>b</sup> College of Biology and the Environment, Nanjing Forestry University, Nanjing 210037, China

<sup>c</sup> University of Chinese Academy of Sciences, Beijing 100049, China

<sup>d</sup> School of Freshwater Sciences, University of Wisconsin-Milwaukee, 600 E Greenfield Ave., Milwaukee, WI 53204, USA

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## ABSTRACT

Stability of colloidal particles contributes to the turbidity in the water column, which significantly influences water quality and ecological functions in aquatic environments especially shallow lakes. Here we report characterization, origin and aggregation behavior of aquatic colloids, including natural colloidal particles (NCPs) and total inorganic colloidal particles (TICPs), in a highly turbid shallow lake, via field observations, simulation experiments, ultrafiltration, spectral and microscopic, and light scattering techniques. The colloidal particles were characterized with various shapes (spherical, polygonal and elliptical) and aluminum-, silicon-, and ferric-containing mineralogical structures, with a size range of 20–200 nm. The process of sediment re-suspension under environmentally relevant conditions contributed 78–80% of TICPs and 54–55% of NCPs in Lake Taihu, representing an important source of colloids in the water column. Both mono- and divalent electrolytes enhanced colloidal aggregation, while a reverse trend was observed in the presence of natural organic matter (NOM). The influence of NOM on colloidal stability was highly related to molecular weight (MW) properties with the high MW fraction exhibiting higher stability efficiency than the low MW counterparts. However, the MW-dependent aggregation behavior for NCPs was less significant than that for TICPs, implying that previous results on colloidal behavior using model inorganic colloids alone should be reevaluated. Further studies are needed to better understand the mobility/stability and transformation of aquatic colloids and their role in governing the fate and transport of pollutants in natural waters.

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## 1. Introduction

Natural colloids, defined as particles with sizes ranging between 1 nm and 1  $\mu$ m, are ubiquitous in aquatic environments (Buffle and Leppard, 1995; Gibson et al., 2007). Due to the small sizes, these colloidal particles usually have a long residence time and substantially impact transparency and recreational value of waters. The properties of high specific surface area and reactivity endow them strong adsorption potential to many trace elements, including nutrients and pollutants, and as a result, influence their chemical speciation, behavior and environmental fate (Gibson et al., 2007). Moreover, the persistence of aquatic colloids can also cause

potential toxicity to microorganism (Shang et al., 2017) and impacts on aquatic ecosystems (Colman et al., 2014). Owing to the significant influence on the fate and transport of pollutants and on ecosystem balance, characterization of aquatic colloids have received increasing attention over the past years.

Environmental colloidal particles in lake waters originate from either allochthonous or autochthonous sources, depending on human activity and local meteorological/hydrological conditions. Compared with deep lakes, shallow lakes are usually characterized by higher turbidity and colloidal abundance due to high primary production and sediment re-suspension (Xing and Kong, 2007; Zheng et al., 2015). The size, composition, and spatio-temporal distribution of lake water colloids had previously been reported, but mainly focused on inorganic colloidal particles (ICPs) (Chanudet and Filella, 2007, 2009; Filella et al., 2009). However, ICPs in natural waters are usually coated with dissolved organic

\* Corresponding author.

E-mail address: [hcxu@niglas.ac.cn](mailto:hcxu@niglas.ac.cn) (H. Xu).

ligands to form organic-inorganic colloidal particles (Schäfer et al., 2007). In addition, dissolved organic matter in natural waters had been shown to occur mostly in the form of colloids (Guéguen et al., 2006; Guo et al., 2009; Shirokova et al., 2013; Stolpe et al., 2013). Therefore, the organic, inorganic and organic-inorganic colloids collectively constitute the abundance of natural colloidal particles (NCPs) in lake waters. Nevertheless, knowledge on the properties and sources of colloidal particles (including NCPs and ICPs) in lake waters, especially the high-turbidity shallow lakes, is lacking, but vital for understanding the roles of colloidal particles in regulating water quality, ecosystem function and pollutant transport.

The environmental behaviors of colloids in natural waters are controlled by their stability/aggregation propensities, which are highly dependent on environmental conditions. Many parameters, including electrolytes, ionic strength, pH, and natural organic matter (NOM), have been reported to influence the stability of inorganic colloids like kaolinite (Kretzschmar et al., 1998; Aurell and Wistrom, 2000; Sequaris, 2010), montmorillonite (Garcia-Garcia et al., 2007; Sequaris, 2010; Borgnino, 2013), illite and quartz (Jiang et al., 2012). Among them, electrolytes and NOM, including quantity and composition, play critical roles in affecting the stability of colloids (Philippe and Schaumann, 2014). It was generally found that electrolytes (including mono- and divalent) enhanced colloidal aggregation, but presence of NOM can significantly inhibit the aggregation potential of colloids/nanoparticles (Chen et al., 2006; Dong and Lo, 2013). However, the NOM-induced aggregation inhibition can be highly related to its inherent chemical structure and molecular size. For instance, bovine serum albumins were found to be more effective than humic and fulvic acids in stabilizing the manganese dioxide colloids due to the difference in molecular sizes of the NOM (Huangfu et al., 2013). In fact, NOM in aquatic environments is a highly heterogeneous mixture with various organic components and a continuous size spectra (Philippe and Schaumann, 2014; Xu and Guo, 2017), and different functional groups and molecular weight (MW) fractions may interact differently with colloidal particles. Within the bulk NOM pool, the high MW (HMW) NOM fraction has been shown to play a central role in regulating the concentration and speciation, and hence the fate, transport and bioavailability of many trace elements in aquatic ecosystems (Guo et al., 2002; Alasonati et al., 2010). In addition, recent studies also highlighted the influence of NOM on colloidal stability and found the MWs and functionalities of NOM an important parameter in controlling aggregation kinetics and stability for nanoparticles, including silver (Yin et al., 2015), gold (Louie et al., 2015), fullerene (Shen et al., 2015), and ZnO (Kteeba et al., 2017). However, these studies usually selected one or two synthesized or engineered nanoparticles as the model materials, while the NCPs are polyfunctional and contain compounds with various compositions and structures. In addition, it is not clear in the aggregation heterogeneities between the NCPs and ICPs in natural waters, leaving a knowledge gap regarding the aggregation behaviors of colloids (including NCPs and ICPs) in environmentally relevant conditions.

The objectives of this study were to: (1) characterize the physicochemical properties of aquatic colloids, including NCPs and ICPs, in shallow lakes; (2) define the origins of these colloidal particles; (3) explore the NOM- and electrolyte-related aggregation profiles in ambient conditions and reveal the specific MW fractions in NOM matrix that regulate colloidal stability. Both spectral and microscopic techniques were used to characterize the chemical properties of colloidal particles, and a simulation experiment was carried out to quantify the contribution of sediment re-suspension processes to the colloid abundance in lake waters. In addition, ultrafiltration was used to fractionate the bulk NOM into LMW- and HMW-NOM fractions, whose specific effects on colloidal

aggregation under mono- and divalent electrolyte conditions were determined via the dynamic light scattering (DLS) technique.

## 2. Materials and methods

### 2.1. Collection of aquatic colloids and sediment core samples

Lake Taihu, located between 30°55'40"–31°32'58" N and 119°52'32"–120°36'10" E, is one of the largest shallow lakes in China (mean water depth: 1.9 m) with high turbidity and low transparency in the water column (Qin et al., 2007). Another feature of Lake Taihu is that it has two distinctive ecological regions: macrophyte- and algae-dominant regions. Surface water samples were collected from the two regions (Fig. S1 in the Supporting Information, SI) with an average wind speed (based on 30 min of continuous measurement) of 2, 4, 6 and 8 m/s, respectively. After collection, the samples were transported to the lab within 4 h and settled statically. The particles with size less than 1  $\mu\text{m}$  (sedimentation velocity < 0.3 cm/h) were collected by siphon based on Stokes' law. The supernatants were then ultrafiltered using a stirred cell ultrafiltration unit (Amicon, 8500) and a 1 kDa membrane disc (Millipore, PLAC07610). The retentate with colloidal sizes between 1 nm and 1  $\mu\text{m}$  was carefully collected to represent the NCPs. An aliquot of the retentate was further treated with 30% hydrogen peroxide to obtain total ICPs (TICPs) until no residual organic matters can be detected in the retentates (Dumat et al., 1997). It was noted that the TICPs herein included pristine inorganic colloids and the inorganic particles that coated with organic matters in natural waters. Both the NCPs and TICPs collected finally were freeze-dried and stored in the desiccator before analysis.

In addition to the colloidal samples from the water column, sediment core samples were also collected at the same sampling location using a  $\varnothing 110\text{ mm} \times 1500\text{ mm}$  gravity corer (Rigo Co., Ltd., Japan). At least 25 cm of intact sediment cores (2.4 L) were sampled (Fig. S2a in the SI), kept upright and transported to laboratory within 4 h of sampling.

### 2.2. Sediment re-suspension experiments

A Y-shape sediment generation equipment (patent number: CN200410014329.X) was used to simulate the re-suspension process of lake sediment at 2, 4, 6 and 8 m/s, respectively (Fig. S2b in the SI). The system consisted of a series of individual polyethylene tubes with a length of 180 cm and a diameter of 11 cm. Two electric motor-driven propellers were equipped to simulate wind-induced waves: one was placed 120 cm above the sediment surface to mix the water vertically and the other was placed 15 cm above the sediment surface in the incline tube with an angle of 55° to offer sediment re-suspension force. The rotation of the two propellers was controlled by different motors with a frequency ranging from 0 to 20 Hz to simulate various wind speeds (Liu et al., 2015).

Before the re-suspension experiment, the sediment cores were carefully transplanted into lower plexiglass tube of Y-shape apparatus. Simulated lake waters (containing 0.15 mM  $\text{K}^+$ , 2 mM  $\text{Na}^+$ , 0.5 mM  $\text{Ca}^{2+}$ , 0.4 mM  $\text{Mg}^{2+}$ , and 1.5 mM  $\text{HCO}_3^-$ ) (Yan et al., 2014) were added to each tube to obtain a depth of about 1.9 m, the same as the mean depth of Lake Taihu. For each experiment, surface water samples were collected after 5 h of re-suspension process, and the colloidal particles were isolated using the above-mentioned ultrafiltration method. Each re-suspension experiment was conducted in duplicate. The difference in colloidal abundance between lake waters and simulation experiments can be used to quantify the contribution of sediment re-suspension to colloids in the water column.

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