

# Occurrence and removal of acesulfame and sucralose in the drinking water treatment plants along the Yangtze River

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## ABSTRACT

Acesulfame (ACE) and sucralose (SUC) are ideal sewage markers for drinking water. In this paper, 33 samples were collected from eight cities' drinking water treatment plants (DWTPs) along the Yangtze River to analyse the occurrence and removal of ACE and SUC. These two compounds were determined using ultra-high performance liquid chromatography–high resolution mass spectrometry. The limits of detection for ACE and SUC were 1.75 ng/L and 0.03 ng/L, respectively. The results showed that ACE and SUC existed widely in DWTPs along the Yangtze River. The concentration of ACE was 13–320 ng/L and the concentration of SUC was 200–1,592 ng/L. In general, the concentrations of ACE and SUC increased from upstream to downstream but decreased after water treatment procedures at every DWTP. In most DWTPs, a 9–30% removal of ACE and SUC was achieved using traditional chlorination disinfection technology. Ozonation combined with granular activated carbon filtration techniques had a high removal efficiency (63.8% for ACE and 50.2% for SUC).

**Key words** | acesulfame, drinking water, occurrence, sucralose, Yangtze River

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## INTRODUCTION

Acesulfame (ACE) and sucralose (SUC) are the most commonly used artificial sweeteners. They are synthetic or semi-synthetic organic compounds, and are widely used in food, drugs, beverages, and personal care products (Chattopadhyay *et al.* 2014). In recent years, artificial sweeteners have generated increasing research interest in the environment (Brorström-Lundén *et al.* 2008; Buerge *et al.* 2011; Ordóñez *et al.* 2012). Many researchers have found that ACE and SUC are persistent and accumulated in environmental water, thus making them ideal anthropogenic wastewater markers (Buerge *et al.* 2009; Richards *et al.* 2017). Because of the prevalence of ACE and SUC in water, many studies have focused on the health effects caused by them on the human body. It has been shown that the long-term use of artificial sweeteners, even in low doses, may

exacerbate senescence and atherosclerosis (Jang *et al.* 2011). Studies have suggested that SUC might be a key pathogenic factor in inflammatory bowel disease, because it has inhibitory effects on intestinal bacteria, and causes inactivation of digestive protease (Qin 2012). In addition, when glycerol is present in cooking, SUC can generate chloropropanols, which are known as genotoxic, carcinogenic, and tumorigenic compounds (Schiffman & Rother 2013). The ACE sweetener is the precursor of several regulated, known, disinfection by-products (DBPs) and two N-DBPs in the process of disinfection. Moreover, new transformation products (TPs) are produced in the hydrolysis of ACE under light irradiation, and these TPs are more toxic in embryonic development than their original compounds (Li *et al.* 2016). Because ACE and SUC provide wastewater marker functions and may have a negative

impact on health, research on their occurrence and distribution in drinking water is necessary.

Many countries have obtained data on the occurrence of ACE and SUC in environmental water or drinking water (listed in Table 1). In 2007, Swedish scientists investigated SUC occurrence in sewage treatment plants (STPs) and found it was minimally degraded (Brorström-Lundén et al. 2008). Four artificial sweeteners (cyclamate, saccharin, ACE and SUC) were detected in most samples from STPs in Switzerland (Buerge et al. 2011). ACE and SUC have been found in STPs in Spain, the USA, and China (Mawhinney et al. 2011; Ordóñez et al. 2012; Gan et al. 2013). In addition, ACE and SUC have been found to be ubiquitous in tap water and surface water. In the USA, the detection rate of SUC in tap water was 76.5%, with concentrations of 49–2,400 ng/L (Mawhinney et al. 2011). In Switzerland, ACE and SUC were detected in surface water with concentrations of 10–949 ng/L and 10–7,346 ng/L, respectively (Berset & Ochsenein 2012). In China, only the seawater of Hong Kong and some river water and drinking water of Tianjin were studied and reported (Gan et al. 2013; Sang et al. 2014). However, there is little reported information on ACE and SUC in Chinese drinking water systems along the Yangtze River in China.

In addition to studies on the occurrence of artificial sweeteners, the removal efficiencies of STPs have also been investigated. Scheurer et al. (2010) evaluated the treatment effect of four artificial sweeteners in different STP units: cyclamate and saccharin were at almost 100% removal by drinking water treatment, while the removal efficiency of SUC was 8–15%. American researchers investigated the degradation of ACE and SUC in chlorination procedures, and the data showed that ACE could degrade by up to 20%, but SUC degradation had not occurred after 24 h (Soh et al. 2011). In China, Li et al. (2018) investigated the fate of four artificial sweeteners, including ACE and SUC, in five STPs that used typical biodegradation methods. The results showed that ACE was only partly removed after biodegradation, and the anaerobic–anoxic–oxic process achieved the highest removal efficiencies (17.78–32.88%), while granular activated carbon (GAC) was preferable in the removal of SUC. Hongwen Sun studied the occurrence and removal of seven artificial sweeteners in STPs and drinking water treatment plants (DWTPs) (Gan et al. 2012); they found that SUC and ACE showed persistence under microbial degradation, and their removal efficiencies were less than 20%. However, these studies were confined to a city or region, and the results are not sufficiently representative of

**Table 1** | Occurrence of ACE and SUC in different areas

Country	SUC (ng/L)	ACE (ng/L)	Year	Reference
USA	Source water: 47–2,900 Finished water: 49–2,400	–	2011	Mawhinney et al. (2011)
Canada	–	River water: 550–1590 Groundwater: 40–390	2013	Spoelstra et al. (2013)
Finland	River: <200–1,000	River: 28–9,600	2014	Perkola & Sainio (2014)
Germany	Surface water: 60–80	Surface water: >2,000	2009	Scheurer et al. (2009)
Switzerland	Tap water: ND Ground water: ND Surface water: <10–949	Tap water: <10–69 Ground water: <10–50 Surface water: <10–7,346	2012	Berset & Ochsenein (2012)
Spain	Creek: 500 River: 600–4,600	Creek: 400 River: 1,200–53,700	2012	Ordóñez et al. (2012)
Singapore	Ground water: <30 Surface water: <50	Ground water: <1.5–95 Surface water: 5–350	2013	Tran et al. (2013)
Hong Kong	Surface seawater: 200	Surface seawater: 340	2014	Sang et al. (2014)
Tianjin China	River water: 287–311 Tap water: 113–171 Ground water: 12.9	River water: 2,730–4,650 Tap water: 786–824 Ground water: 24.8–45.7	2013	Gan et al. (2013)

a vast country such as China. These limitations apply especially to the Yangtze River, the largest and longest river in China, which provides drinking water for about 400 million people, and flows through 11 provinces and two province-level municipalities.

To address these knowledge gaps, the occurrence and removal efficiencies of ACE and SUC in DWTPs along the Yangtze River were investigated. Besides improving the knowledge of the status of SUC and ACE in drinking water, the results of the study will allow the effects of sewage on drinking water along the Yangtze River to be more clearly identified.

## MATERIALS AND METHODS

### Chemicals and reagents

ACE (1,000 µg/mL, 5 mL) was purchased from Beijing Tanmo Quality Inspection Technology Inc. (Beijing, China); SUC (99.8%) was obtained from the National Institutes for Food and Drug Control (Beijing, China); ACE-d4 (99.0%) and SUC-d6 (99.0%) were obtained from Toronto Research Chemicals Inc. (Toronto, Canada); methanol of LC-MS grade was purchased from Fisher Scientific Inc. (Venezuela); formic acid (FA, 98%) of MS grade was purchased from Sigma-Aldrich (Switzerland); pure water was purchased from Watsons (Guangzhou, China).

All the standard stock solutions were individually dissolved in methanol at 500 mg/L, and stored at  $-20^{\circ}\text{C}$ . The working standard solutions were diluted with 50% methanol containing 0.1% FA and stored at  $4^{\circ}\text{C}$ .

### Analytical methods

The separation of ACE and SUC was performed with an UltiMate 3000 series liquid chromatograph (Thermo Scientific, USA). The chromatographic column was the Dikma C18 column ( $100 \times 3$  mm,  $3\ \mu\text{m}$ ). The gradient elution conditions were: mobile phase A was water containing 0.1% FA and B was methanol with 0.1% FA; gradient: 0–2.5 min, 2–95% B; 2.5–5.5 min, 95% B; 5.5–6 min, 95–2% B; 6–12 min, 2% B; flow rate = 0.4 mL/min; column temperature =  $30^{\circ}\text{C}$ ; injection volume was set to 100 µL.

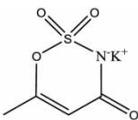
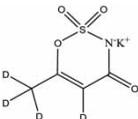
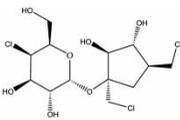
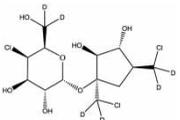
The MS analysis was performed with a Q-Exacte Quadrupole-Orbitrap High-Resolution Mass Spectrometer (Thermo Scientific, USA). The scanning modes were negative target-SIM mode during quantitative analysis and negative PRM mode in qualitative analysis. The source parameters were as follows: heater gas: 42 L/h, aux gas: 15 L/h, spray voltage: 3.5 kV, heater temperature:  $320^{\circ}\text{C}$ , capillary temperature:  $350^{\circ}\text{C}$ . The quantitative and qualitative analysis parameters are summarized in Table 2 and in Figure S1 in the supporting information (available with the online version of this paper).

All the samples were prepared with 0.22-µm filters (hydrophilic, Merda) and were introduced to ultra-high-performance liquid chromatography (UHPLC) by the automatic sampler. ACE-d4 (500 ng/L) and SUC-d6 (1,000 ng/L) were added to the water samples before filtration, for the isotopic internal standard of ACE and SUC quantification, respectively. Each sample was repeated three times and the average value was calculated. The standard solutions were made by 50% methanol containing 0.1% FA and the isotopic internal standards were added. Then, the standard curve was drawn using the concentration ratio as the *x*-axis, and the peak area ratio as the *y*-axis (Figure S4). The linear ranges were 10–1,000 ng/L and 10–2,000 ng/L for ACE and SUC, respectively, and the correlation coefficients were all greater than 0.99. The performance of the proposed method was executed and the results are listed in Table S1. From the results it was known that the method was accurate and concise, and was suitable for the quantification of ACE and SUC in the actual water samples. (Figure S4 and Table S1 are available online.)

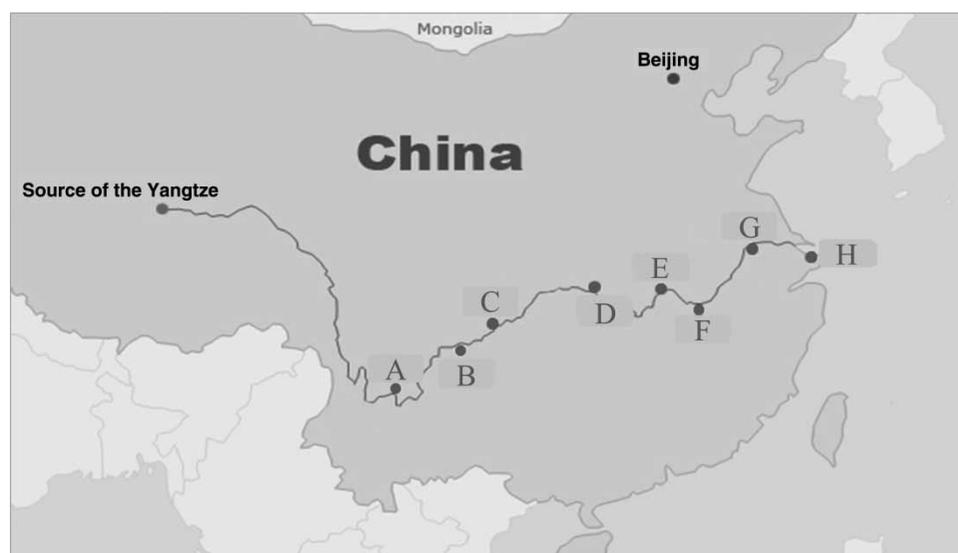
### Sample collection

A total of 33 samples were collected from eight DWTPs of eight cities along the Yangtze River in September 2017 (sampling sites are shown in Figure 1). These sampling sites cover five provinces and two province-level municipalities. Most of the DWTPs sampled apply conventional water treatment methods (coagulation, precipitation, filtration, and chlorination). Four samples were obtained from every site and were named after the alpha code of the sampling city, and then numbered 1, 2, 3, and 4 for surface water (grabbed from the river directly), input water (raw water about 15 m deep), output water (treated water) and users' domestic water (from residents at about 5 km from the water plant). Sampling site D is different from the

**Table 2** | Chemical structure and analysis parameters of the sweeteners

Compound	Retention time	Structural	Parent ion ( <i>m/z</i> )	Daughter ion ( <i>m/z</i> )
ACE	3.20 min		*162	77.9 82.0
ACE-d4	3.20 min		*166	77.9 86.0
SUC	3.56 min		395 397 *441 443	359.0 361.0 395.0 359.0 397.0 361.0
SUC-d6	3.57 min		401 403 *447 449	365.0 367.0 401.0 365.0 403.0 367.0

\*Quantitative ion of the target compound.

**Figure 1** | The sampling points along the Yangtze River.

others because the source water is one branch of the Yangtze River. In city H, a sample of advanced treatment (ozonation combined GAC filtration) was obtained and labeled as H5. All the water samples were put into ice bags immediately after collection, transported to the laboratory, and stored in a 4 °C refrigerator. Ultrapure water was used as a blank sample and treated with the water samples from the DWTPs.

## RESULTS AND DISCUSSION

### Development of the analytical method

The UHPLC mobile phase gradient was adjusted based on the approach of Wu *et al.* (2014) to make the elution completely separate. After optimizing the high-resolution

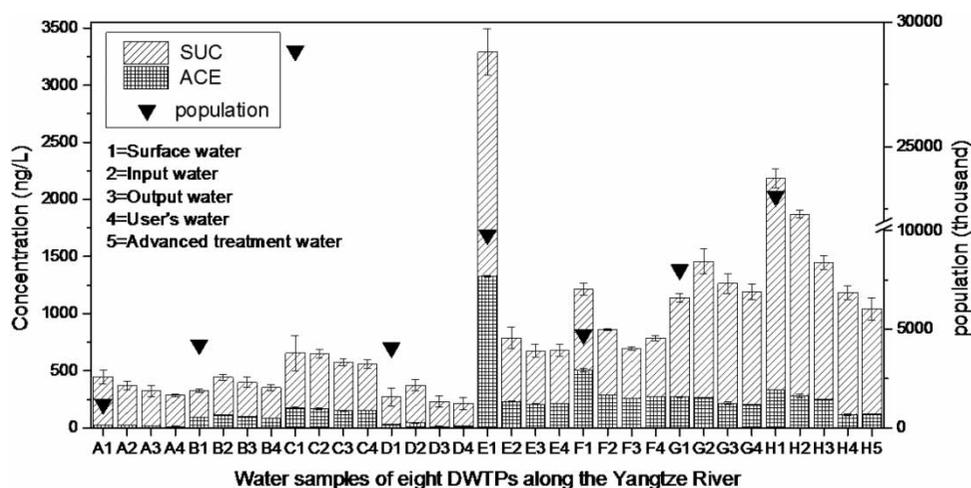
mass spectrometry (HRMS) parameters (Figure S2), the FA adducts  $[M + HCOO]^-$  were chosen as quantitative ions of SUC by obtaining a three-fold signal intensity to the previous quantitative ions of  $[M-H]^-$  (Figure S3). (Figures S2 and S3 are available with the online version of this paper.) Based on the signal-to-noise ratio of 3, the limits of detection (LODs) for ACE and SUC were determined as 1.75 ng/L and 0.03 ng/L, respectively. Comparing with the previous methods requiring solid-phase extraction pretreatment (Mawhinney *et al.* 2011), or large volume injection of 500  $\mu$ L (Wu *et al.* 2014), this proposed method has the advantages of rapid and easier operation, and lower LOD.

### Occurrence of ACE and SUC along the Yangtze River

The concentrations of the 33 water samples were determined using the proposed analytical method. Figure 2 shows the occurrence of ACE and SUC along the Yangtze River, and Table 1 provides data from other studies. The concentrations of ACE are 13–280 ng/L and the concentrations of SUC are 200–1,592 ng/L. The concentrations of ACE in surface water range from 24 ng/L to 507 ng/L in the present study, which is similar to the levels reported from Singapore (5–350 ng/L) (Tran *et al.* 2013) and Hong Kong (340 ng/L) (Sang *et al.* 2014), but much lower than that from Switzerland (10–7,346 ng/L) (Berset & Ochsenein 2012). The ACE concentrations in tap water in the present study are lower than that of Tianjin. The SUC concentrations observed (240–1,850 ng/L) in this paper

are slightly lower than those from the United States (49–2,400 ng/L) (Mawhinney *et al.* 2011), and higher than those from Germany (60–80 ng/L) (Scheurer *et al.* 2009) and Singapore (<50 ng/L) (Tran *et al.* 2013). The lower concentrations of ACE and SUC in surface water in Germany and Switzerland may be the result of their efforts to improve environmental water quality, which also makes the concentrations of ACE and SUC in tap water and groundwater relatively low (Meng 2007). In Singapore and Hong Kong (Sang *et al.* 2014), sewage is discharged into the deep sea after centralized treatment of the waste water, and water is imported from abroad (Meng 2007). These factors may be the reason for the lower concentrations of ACE and SUC in water samples from Singapore and Hong Kong.

Figure 2 shows that the concentrations of ACE and SUC gradually increase in the downstream direction. This trend is particularly noticeable in the surface water samples. The persistence and accumulation of ACE and SUC in aquatic environments, and sewage discharge in the cities along the Yangtze River, may be the reason for this trend (Scheurer *et al.* 2010). But there are two abnormal phenomena in the DWTP sampled of city D and city E. The concentrations of ACE and SUC are all lower than those of the adjacent city C and E but close to that of the uppermost city A. It is speculated that the source waters of city E and city A were less affected by sewage discharge, because the source water of city D is a branch of the Yangtze River, which originates from the mountains nearby and flows through fewer industrial and residential areas. So it does for city A,



**Figure 2** | The occurrence of ACE and SUC in DWTPs along the Yangtze River and the population levels of eight cities.

where the source water is close to the cradle of the Yangtze River. As for the highest value in the surface source water, for city E, this implies that some sewage possibly is discharged into the surface water, whereas the input water at a depth of 15 m is not much affected.

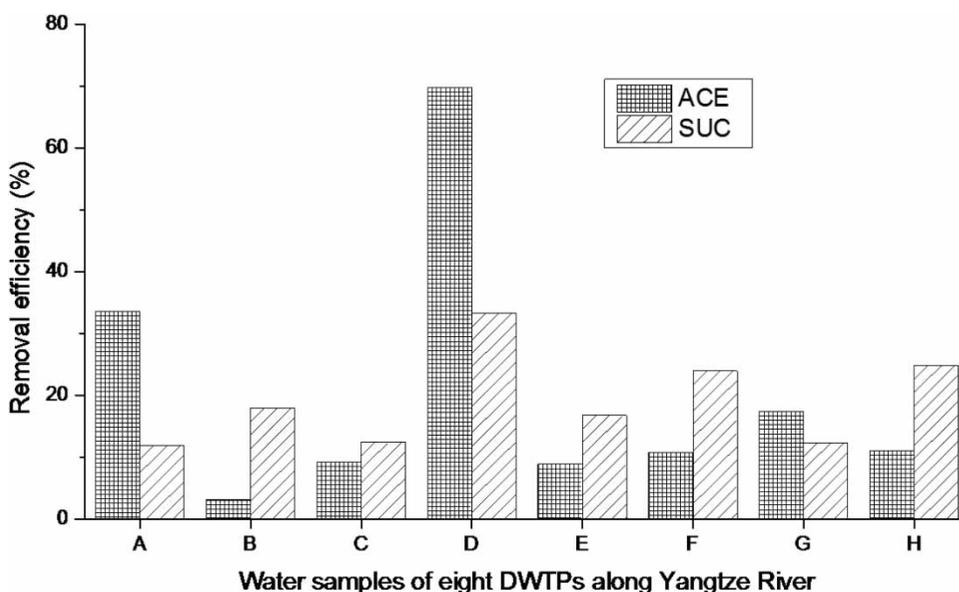
### Correlation analysis between ACE and SUC concentrations and population levels

According to the previous literature, ACE could also be used as a population marker. [Buerge \*et al.\* \(2009\)](#) determined the concentrations of ACE in four rivers and nine lakes in Switzerland, and reported that concentrations of ACE were proportional to the ratio of the population in the catchment area per throughflow of water. [Müller \*et al.\* \(2011\)](#) collected water samples from 44 locations with populations from 0 to 1.8 million, and found the correlation of ACE and population was 0.94. However, different results were obtained in this paper. The values of ACE and SUC were fitted with the corresponding cities' population with Excel (Microsoft Office 2016, as depicted in Table S3), and also the correlations between the artificial sweeteners and the ratio of population per water flow were made (as shown in Figure S5). (Table S3 and Figure S5 are available online.) The results showed that there was poor correlation between

them. There are two reasons for the difference. (1) Sample module. In the previous study, the water sampling locations were decentralized and less connected. But in this paper, the water samples were obtained with the river flow from upstream to downstream. (2) The characteristic of persistence and accumulation of ACE and SUC. From [Figure 2](#) it can be seen that the concentrations of ACE and SUC increase gradually with the flow, but the populations of the cities along the river do not. For instance, the population of the downstream city F is similar to the upstream city B, but the ACE and SUC concentrations are much higher. For city C, China's most populous city, the values of ACE and SUC are far below those of the downstream cities. Above all, it is concluded that the population marker study does not suit a long-distance flowing river. However, the correlation between ACE and SUC is 0.6971, which suggests that they may have similar pollution pathways and mechanisms.

### Removal of ACE and SUC during drinking water treatment

[Figure 2](#) shows that ACE and SUC in output water are apparently lower than in input water for every DWTP, which implies that part of them should be removed during the treatment process. The removal efficiency is presented in [Figure 3](#),



**Figure 3** | The removal efficiencies of ACE and SUC in DWTPs in the eight cities investigated.

and the calculation formula for removal efficiency is:

$$R = \frac{C_i - C_o}{C_i} \times 100\% \quad (1)$$

where  $R$ : removal efficiency,  $C_o$ : output concentration,  $C_i$ : input concentration.

Figure 3 shows that the removal is similar in the DWTPs of the downstream cities, and the median value is about 11% for ACE and 17% for SUC. The SUC removal data are consistent with the results obtained in the USA (Soh *et al.* 2011) for the laboratory simulation of SUC removal using chlorination (Li *et al.* 2018). As for ACE, the reduction after the drinking water treatment procedure may be caused by the transformation of ACE to some DBPs (Li *et al.* 2017). Studies have shown that ozonation combined with GAC filtration can effectively remove ACE and SUC in water (Scheurer *et al.* 2010). Our analysis of sample H5 confirms this and shows that ACE and SUC removal efficiencies reach 63.8% and 50.2%, respectively.

## CONCLUSIONS

In this paper, an economical, rapid, and convenient analytical method was developed to directly quantify ACE and SUC in drinking water using UHPLC-HRMS. The water samples were collected from the DWTPs of eight cities along the Yangtze River, and the occurrence and removal of the two compounds were investigated. The results show that ACE and SUC are ubiquitous with concentrations of 13–280 ng/L for ACE and 200–1,592 ng/L for SUC, and increase in the downstream direction. In general, the median removal rate is 11% for ACE and 17% for SUC for DWTPs using traditional chlorination, but increases to 63.8% for ACE and 50.2% for SUC when an advanced ozonation and GAC treatment is used.

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