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Inhibition and decomposition effects and mechanisms of gallic acid on silica scale

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ABSTRACT

Silica scale is particularly difficult to remove from water systems due to its complex composition and hard texture. We investigated the effect of gallic acid (GA) on silica scale formation through interactions between GA and polysilicic acid. Silicic acid speciation result shows that GA increases the concentration of the soluble silicic acid by the formation of water soluble Si-GA complexes. The complexes prevent the formation of silica scale by inhibiting the polymerization of silicic acid. The particle size distribution analysis showed that GA adsorbed on the surface of polysilicic acid particles with the subsequent formation of complexes, reducing the particle size of polysilicic acid by decomposition. Gel chromatography showed that GA decomposed smaller polysilicic acid sizes to promote decomposition reactions (polysilicic acid becoming monosilicic acid, P→M), inhibiting silica scale formation. GA was effective as an inhibitor and in the decomposition of silica scale, presenting clear potential for application to real-world water systems.

Keywords: Silica scale; Polysilicic acid; Inhibition; Decomposition; Gel chromatography

1. Introduction

The formation of mineral scale in pipelines, wells, heat exchangers, and membranes is a serious problem hindering the widespread utilization of water resources and seawater desalination [1–4]. Once scale begins to form, it significantly limits production efficiency and shortens the lifetime of equipment due to blockages and corrosion of the pipeline, membrane, etc. [5–7]. Therefore, the operations have to be stopped regularly for mechanical washing or chemical cleaning after running for a period of time, causing equipment damages and economic losses [8,9]. Calcium carbonate, magnesium carbonate, calcium sulfate, and silica are common mineral scales [10]. Among them, silica scale, the general term for insoluble silicate, silica gel, and silica particles, has become the most difficult to remove at present due to its hard texture and insolubility in ordinary acid and alkali [11,12]. Compared with carbonate scale, silica scale has a more complex composition and formation mechanism (Fig. 1), therefore, carbonate scale treatment methods commonly used lead to unsatisfactory results when used with silica scale [10,13].

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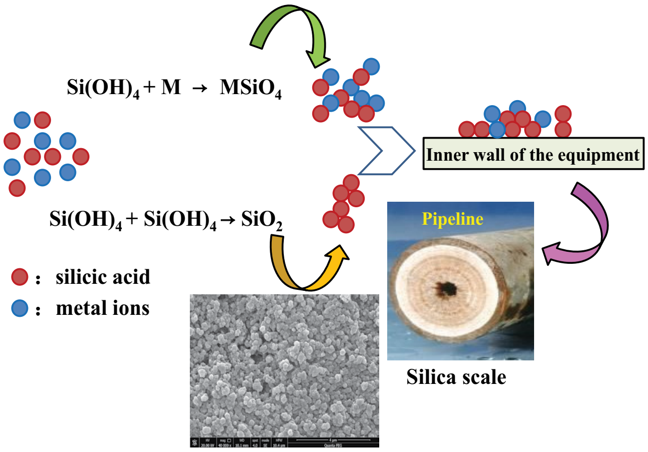

Fig. 1. Schematic diagram of silica scale formation process.

Hydrofluoric acid, hydrofluoric amine, and sodium hydroxide are the most common silica scale cleaning agents, but these agents have a tendency to corrode equipment and may produce a secondary scale of $Ca(OH)_2$ or $Mg(OH)_2$ [14,15]. Therefore, organic compounds are used as scale inhibitors and chemical cleaning to prevent or control silica scale formation by most industries at present [10,16–18]. The inhibitors used on a large scale are commonly organic macromolecules containing hydroxyl, carboxyl, amine, or other functional groups, which chelate, disperse, or otherwise interfere with crystal growth [10,19,20]. For silica scaling, the inhibition mechanisms mostly include interfering with the silica polymerization process and dispersing colloidal silica particles [21]. Inhibition effects of some cationic polymers, such as polyaminoamide (PAMAM) dendrimers with $-NH_2$ termini, and phosphine carboxylic acid salts (DP6070) on silica scale have been reported [10,13,16–18]. However, scale inhibitors containing nitrogen and phosphorus may cause eutrophication in water bodies, the phosphorus-free, nitrogen-free, biodegradable, and cost-effective inhibitors have received more concerns considering environmental impact.

Gallic acid (GA) is a natural polyphenolic substance with one carboxyl group and three o-hydroxyl groups. It can be widely found in plants and characterized as water-soluble, low-cost, and free from nitrogen and phosphorus [22]. It has similar functional groups with common scale inhibitors, suggesting its potential use as a silica scale inhibitor. In addition, the gallic acid modified resin has an ideal adsorption capacity for various forms of silicic acid and can effectively prevent the formation of silica scale [22]. Moreover, GA and its derivatives are widely used as nanoantioxidant materials, engineered by covalently grafting GA onto SiO_2 nanoparticles, thus demonstrating strong interactions between GA and silica [23]. However, the interaction details between GA and silicic acid are rarely reported, notably, the effect of GA on silicic acid precipitation. The polymerization of silicic acid is an important part of the formation of silica scale, thus revealing the effect of GA on silicic acid polymerization to provide valuable information regarding inhibiting the formation of silica scale. Therefore, in this study, we assessed the influence of GA on the formation of silica scale to reveal the relevant influence mechanisms and provide new and valuable information for the future development of improved silica scale inhibitors and washing agents.

2. Experimental design

2.1. Materials and sample solutions

All chemicals used were analytical reagents purchased from different companies. All solutions used were prepared with ultrapure water (Milli-Q SP System, Millipore). The 0.1 M (M: mol/L) silicic acid stock solution was prepared by dissolving sodium metasilicate ($Na_2SiO_3 \cdot 9H_2O$, Wako Junraku Co., Ltd., Japan) in a 0.1 M of NaOH solution with a concentration determined by inductively coupled plasma atomic emission spectrometry (ICP-OES OPTIMA 8000, PerkinElmer, USA). GA (Kulaibo Technology, Beijing) was used in the solid-state. Each solution was prepared to its desired concentration by diluting the stock solution with water before use. The concentration of silicic acid was expressed as Si mg/L. Sephadex G-100 and Blue Dextran 2000 (Pharmacia, Uppsala, Sweden) were purchased from GE Healthcare. A 0.4% Blue Dextran 2000 solution, containing 0.1 M NaCl (pH 2) was used for measurement of the void volume of the gel bed in gel chromatography. The 0.1 M NaCl solution (pH 2) was used as an eluent. During the experiment, the pH of the solution was adjusted by diluted HCl or NaOH solution.

2.2. Effect of GA on silicic acid polymerization

To evaluate the effect of GA on the polymerization of silicic acid, the sodium silicate solution (400 mg/L, 1,500 mL) was adjusted to pH 8 ± 0.2 and stirred vigorously by a magnetic stirrer for 5 h. Afterward, this solution was divided into four 250 mL portions, to which were added different amounts of solid state GA to maintain concentrations of 0, 40, 160, and 400 mg/L. After the GA was thoroughly dissolved, the pH of the solutions was reset to 8 ± 0.2 and these were continuously stirred. At certain time intervals, aliquots of the suspensions were removed and filtered using 0.45 μm membrane filters. The concentrations of monosilicic acid and total silicic acid in the filtrate were determined by UV-Vis spectrophotometry (Shimadzu UV-3600, Japan) and ICP-OES, respectively. The concentrations of the different forms of silicic acid were then calculated as follows:

Concentration of monosilicic acid: $C_{H_4SiO_4}$ (calculated from spectrophotometry):

Concentration of polysilicic acid <0.45 μm size:

$$C_{<0.45\,\mu m\ PolySi} = C_{ICP\text{-}OES} - C_{H_4SiO_4} \tag{1}$$

Concentration of polysilicic acid > 0.45 μm size:

$$C_{>0.45\,\mu m\ PolySi} = C_0 - C_{ICP\text{-}OES} \tag{2}$$

Initial concentration:

$$C_0 = C_{H_4SiO_4} + C_{<0.45\,\mu m\ PolySi} + C_{>0.45\,\mu m\ PolySi} \tag{3}$$

2.3. Effect of GA on particle-size variation during silicic acid polymerization

Polysilicic acid with different degrees of polymerization was prepared in advance by the same methods in Section 2.2. After polymerization for 5 h, this was also divided into four 250 mL portions, different amounts of solid state GA were added, and all portions were stirred for 8 h. At certain time intervals, aliquots of the suspensions were removed, and the polysilicic acid particle size distribution was directly measured using a laser diffraction particle size analyzer (MicroTrac S3500, USA) with 166 detector channels and 0.01–2,800 μm, respectively.

2.4. Separation of different silicic acid forms by gel chromatography

Gel chromatography separates different molecular sizes or shapes components using a gel-filled column, quite suitable for the separation of monosilicic and polysilicic acids with different particle sizes [24,25]. Polymers with large volumes were eluted by eluent earlier, as they could not enter the gel; the larger the volume, the earlier the outflow. However, small molecules flowed out later due to the gel particles strong permeability. In order to investigate the detailed antiscaling mechanism of GA on the formation of silica scale, different forms of silicic acid formed in the absence and presence of GA solution were separated by gel chromatography.

2.4.1. Column filling of gel chromatography

Sephadex G-100 gel was suspended in a 0.1 M NaCl solution (pH = 2) for 24 h and allowed to swell fully, then was loaded into a medium-pressure special gel chromatography column with a 45 cm × 1.6 cm I.D. and a porous polystyrene disk at the bottom. The peristaltic pump speed was adjusted to 2 rpm, and the column was eluted under pressure until the column height was stable. The void volume (V_0) of the gel bed in gel chromatography was 34 mL, which was measured by introducing of the 0.4% Blue Dextran 2000 solution into the column (V_0 was measured after column height was stable).

2.4.2. Separation experiment

Polymerization experiments using silicic acid with or without GA were carried out. For a single silicic acid solution, the 400 mg/L (50 mL) of silicic acid solution was adjusted to pH 8 ± 0.2 and stirred vigorously by a magnetic stirrer for several hours. At regular time intervals (0.5, 1, 5, 10 h), 2 mL of suspension was removed and put on the top of the gel chromatography column, then eluted with 0.1 M NaCl solution (pH = 2) to separate silicic acids based on their size. Meanwhile, the 2 mL of effluent was collected in each fraction using an automatic fraction collector (ADVANTEC CHF 121SA), and the Si concentration in each fraction was measured by ICP-OES. For the presence of GA solution, the polysilicic acid with different degrees of polymerization was prepared (four 50 mL) in advance by same method with the single silicic acid solution for 5 h. Afterward, a certain amount of solid state GA was added into each solution, maintaining the concentration of GA in solution was 0, 40, 160, and 400 mg/L, respectively. The separation method was the same as the single silicic acid solution.

3. Results and discussions

3.1. Effect of GA on silicic acid polymerization

In natural waters, the concentration of silicic acid is less than 10 mg/L, however, it increases with cyclical reuse of water in industrial systems. When the concentration of monosilicic acid is supersaturated to the solubility of amorphous silica, polymerization of silicic acid occurs immediately, forming nanometer-sized polysilicic acid particles. The particles adhere to infrastructure to form silica scale [6,26]. Fig. 2 shows variations in different forms of silicic acid concentration in the absence and presence of GA solutions. Monosilicic acid and both polysilicic acids were already present at 0 h, reflecting prepolymerization occurring during the initial 5 h (Fig. 2a). Subsequently, the monosilicic acid concentration decreased slowly over time and the polysilicic acid concentrations increased, indicating the polymerization continued. However, the variation trend of the two polysilicic acid sizes was not clear.

When GA was added, the initial and subsequent monosilicic acid concentrations increase, indicating that part of the polysilicic acid decomposed to soluble silicic acid by GA addition (Fig. 2b). Disodium 4,5-dihydroxy-1,3-benzenedisulfonate (tiron), a derivative of catechol, interacts with silanol groups in the monosilicic acid to form a water–soluble silicic acid organic compound complex in an aqueous solution under near-neutral pH [27]. GA is also a derivative of catechol and has a similar molecular structure to tiron. It suggests that the three o-hydroxyl groups on a benzene ring also can interact with monosilicic or polysilicic acid to form water–soluble compounds. Based on the concentration of monosilicic acid in pure water under stable temperature was constant [28], in the presence of GA, the concentration of soluble silicic acid in solution equals the sum of monosilicic acid and the silicic acid-GA complex [Eq. (4)]. Moreover, the concentrations of both forms of polysilicic acid were lower than the non-GA case, indicating that both oligomers and polysilicic acid >0.45 μm can be decomposed by GA. This indicates that GA can retarding the polymerization of silicic acid and prevents the formation of silica scale.

$$C_{\text{soluble silicic acid}} = C_{H_4SiO_4} + C_{\text{Si-GA}} \tag{4}$$

where $C_{\text{Si-GA}}$ is a concentration of silicic acid-GA complex. The concentration of monosilicic acid in pure water under constant temperature was constant [28].

To further clarify the inhibition effect of GA on silica scale formation, silicic acid polymerization experiments were carried out in the presence of GA at concentrations from 0–400 mg/L (Fig. 3). As shown in Fig. 3, compared with the absence of GA, the concentrations of three forms of silicic acid conspicuously changed in all mixed solutions with GA, indicating that GA had a significant impact on the polymerization of silicic acid. As shown in Fig. 3a, compared with the absence of GA, the soluble silicic acid

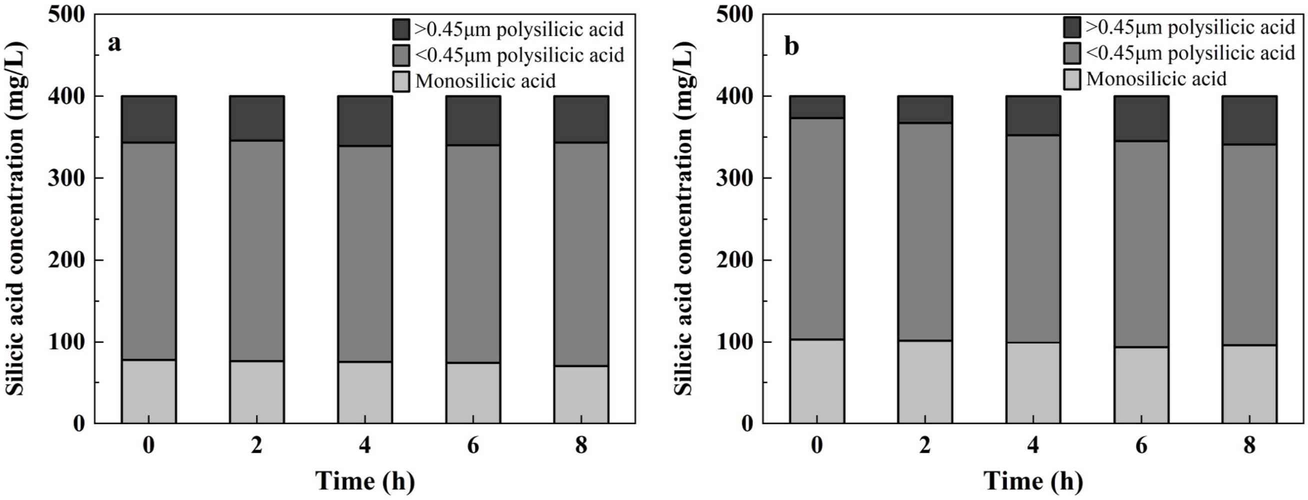

Fig. 2. Variations in different forms of silicic acid concentration over time in the (a) absence and (b) presence of GA (160 mg/L).

concentration was clearly increased with increasing the GA concentration, but both forms of polysilicic acid concentration were decreased, further indicating that polysilicic acid with different degrees of polymerization can be decomposed by GA in a short time. The concentration of polysilicic acid >0.45 μm increased with reaction time (Fig. 3b and c), while that of polysilicic acid <0.45 μm decreased, suggesting that some of the latter grew into the former. There was no correlation between the two forms of silicic acid concentration (Fig. 3d), indicating that small polysilicic acid both decomposed and polymerized. Therefore, the system containing GA was in a dynamic equilibrium of polymerization and depolymerization, but the decomposition rate was faster than the polymerization rate.

3.2. *Effect of GA on the growth of polysilicic acid particles*

Polymerization of silicic acid is begun by the condensation reaction between two monosilicic acid molecules, forming the dimer Si–O–Si. This continues to react with monomers or other dimers to grow silica particles of a certain size [24,29]. To confirm the decomposition effect of GA on the formation of silica, the silica particle size distribution in the presence and absence of GA at different concentrations was measured by a laser diffraction particle size analyzer. The main particle size of polysilicic acid ranged from 60–80 μm after 3 h polymerization, gradually increasing over time (Fig. 4). After 13 h, the size of polysilicic acid reached 160–180 μm, indicating that the polymerization reaction or aggregation of silica particles continued to occur in the single silicic acid solution, leading to the growth of large particles. This result is consistent with a report by Heuvel et al. [5,26]. In Fig. 3, the concentration of polysilicic acid with a size less than 0.45 μm was highest. The particle sizes measured here were mainly produced with a size larger than 0.45 μm and the polysilicic acid with a size less than 0.45 μm cannot be detected during this time due to the channel accuracy of the analyzer.

In Fig. 5a, compared with the absence of GA (0 mg/L), the polysilicic acid particle sizes gradually decreased with the increasing of GA concentration, and the main particle size decreased significantly. This finding indicates that the polysilicic acid particles are decomposed by GA (Fig. 5a). This result is different from other studies [26], but was consistent with that of the tiron [24]. Although both phthalate and GA are derivatives of catechol, the results prove that phthalate was different from GA when they react with silicic acid due to different functional groups. Hence, for scale inhibitors, the functional groups in the molecules are an important factor.

Fig. 5b shows that the decomposition effect of GA increased with reaction time increases, consistent with Fig. 3. The peak at <5 μm particle size was detected after adding GA, indicating that GA had a strong decomposition effect on silica. Compared with the absence of GA, the main particle size decreased rapidly within 0.5 h and then gradually increased over time. The main particle size of polysilicic acid in the mixed solutions fluctuated in the range of 30–55 μm at 8 h (equivalent to 13 h after addition of GA), but this was smaller than in the absence of GA solution, indicating that GA could both decompose macropolymers and inhibit the growth of polysilicic acid particles. These results suggest that the solubility and the decomposition of silica are both promoted by GA.

3.3. *Separation of different forms of silicic acid by gel chromatography*

The gel chromatograms for silicic acid in the absence of GA solution (Fig. 6a) showed two distinct peak clusters: the one on the right side corresponded to monosilicic acid and the other on the left side corresponding to polysilicic acid particles. Different eluent volumes are corresponding to different degrees of polymerization, and the peak area was proportional to the content of the substance. The peak top of polysilicic acid gradually shifted to the left with a longer polymerization time, from 72 mL at 0.5 h to 34 mL at 10 h, indicating that the size of polysilicic acid gradually increased. However, the peak top of the monosilicic acid in each curve was the same (96 mL), indicating that the size

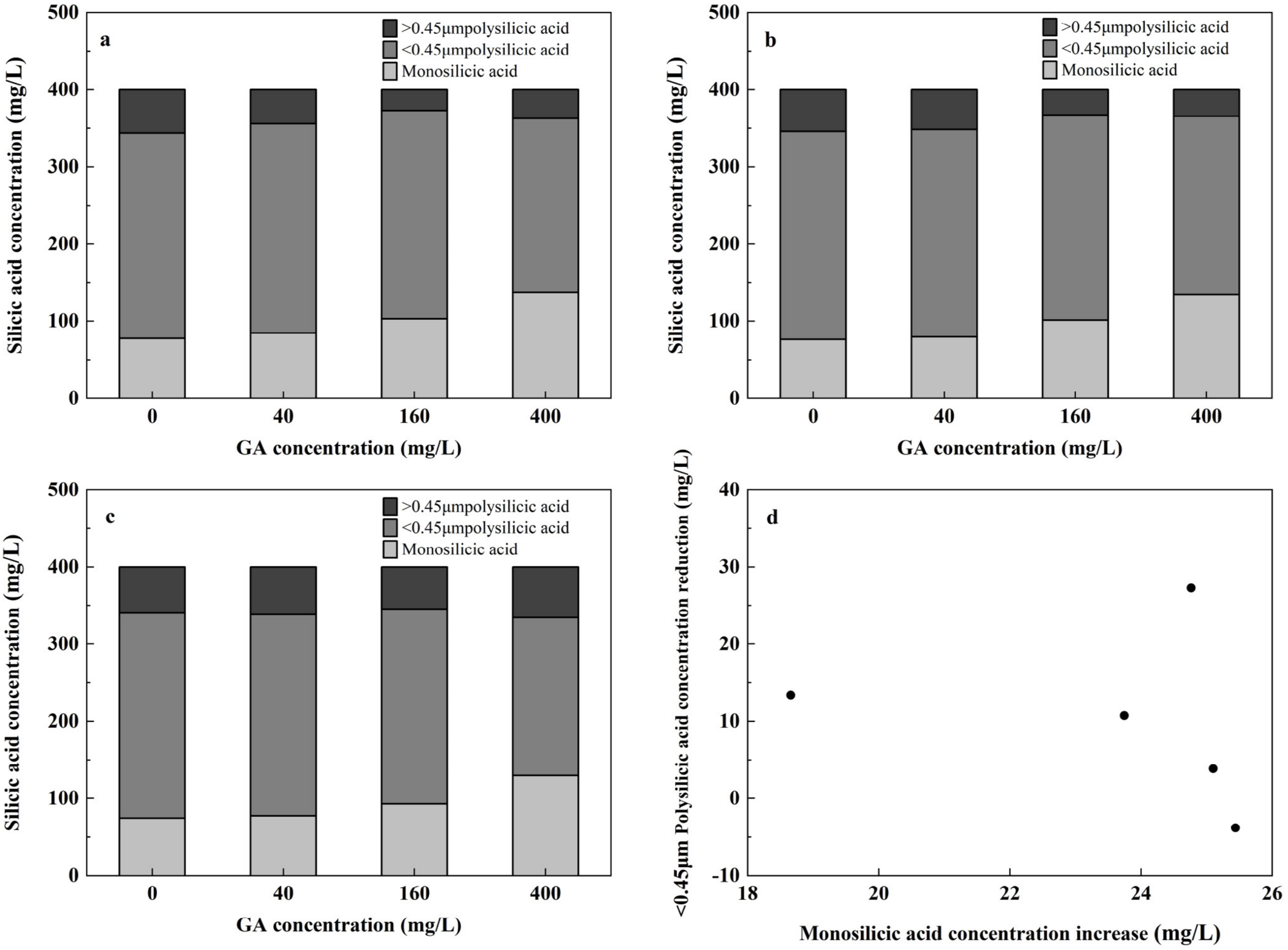

Fig. 3. Variations in concentrations of different silicic acid forms in the presence of different GA concentrations at (a) 0 h, (b) 2 h, and (c) 6 h; (d) plots the relationship between increase the monosilicic acid concentration and decrease the <0.45 μm polysilicic acid concentration at 6 h.

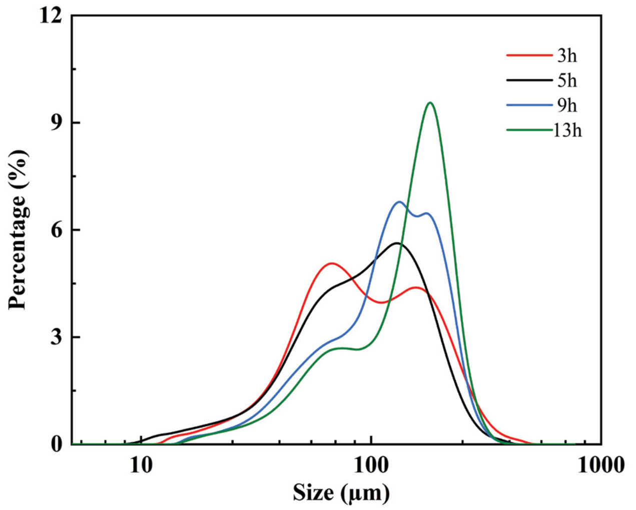

Fig. 4. Variations in the size distributions of silica particles in the absence of GA solution.

of monosilicic acid was the same. On the other hand, the area of the right peak decreased gradually, while the area of the left peak increased, indicating that the monosilicic acid gradually polymerized to form polysilicic acid.

In the presence of GA solution (Fig. 6b), the peak on the right side was due to the silicic acid-GA complex in addition to free monosilicic acid, since the complex elutes plotted at the same position as monosilicic acid. The peak area on the right side increased significantly with increasing GA concentration, while that on the left decreased, indicating that polysilicic acid can be dissolved into silicic acid by GA. Compared with the absence of GA (Fig. 6a), the peak intensity corresponding to the effluent volume of 50–70 mL was significantly reduced, indicating that the increase in the peak intensity of soluble silicic acid was mainly caused by the decomposition of polysilicic acid with smaller particle size, consistent with Fig. 4.

Silicic acid polymerization can be divided into three steps: (1) a reaction between monosilicic acids (M-M reaction); (2) a reaction between monosilicic acid and polysilicic acid (M-P reaction); and (3) a reaction between polysilicic

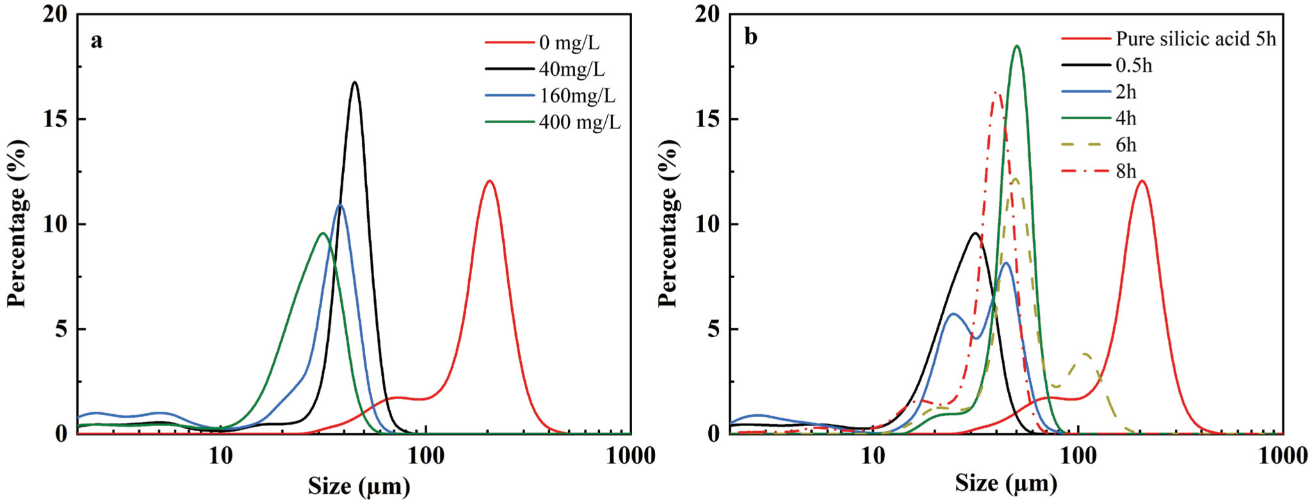

Fig. 5. Variations in silica particle size distributions in solutions: (a) at 0.5 h by different GA concentration and (b) in the 400 mg/L GA solution by time.

acids (P-P reaction, aggregation), which may lead to the formation of a microgel [Eqs. (5)–(7)] [24,29]. Further, silicic acid polymerisation, nucleation, and growth are enhanced at slightly alkaline pH, elevated temperature, medium to high ionic strength (especially the presence of Al and Fe), and high total initial concentrations [28,30–32].

$$Si(OH)_4 + Si(OH)_4 \rightarrow (OH)_3Si{-}O{-}Si(OH)_3 \text{ (M-M)} \quad (5)$$

$$(OH)_3Si{-}O{-}Si(OH)_3 + Si(OH)_4 \rightarrow (OH)_3Si{-}O{-}Si(OH)_2{-}O{-}Si(OH)_3 \text{ (P-M)} \quad (6)$$

$$(OH)_3Si{-}O{-}Si(OH)_3 + (OH)_3Si{-}O{-}Si(OH)_3 \rightarrow (OH)_3Si{-}O{-}Si_n(OH)_m{-}O{-}Si(OH)_3 \text{ (P–P)} \quad (7)$$

According to the gel chromatography results, GA increased the concentration of soluble silicic acid by decomposition of polysilicic acid, indicating that GA inhibited the P-M and P-P reactions. As there is a strong affinity between GA and various forms of silicic acid [2], we speculate that GA first adsorbs onto the surface of polysilicic acid to form a surface complex and it enters the solution in the form of a soluble complex, then the polysilicic acid was gradually decomposed into soluble silicic acid by this complex. The schematic diagram of this antiscaling mechanism (Fig. 7) shows that GA not only forms complexes with monosilicic acid to improve the solubility of silica, but can also be adsorbed onto the surface of silica particles to decompose the silica by the “corrosive effect”.

4. Conclusions

The natural organic compound GA is widely present in plants and can be used to inhibit and decompose silica scale. Dissolved GA reacts with monosilicic acid in water to

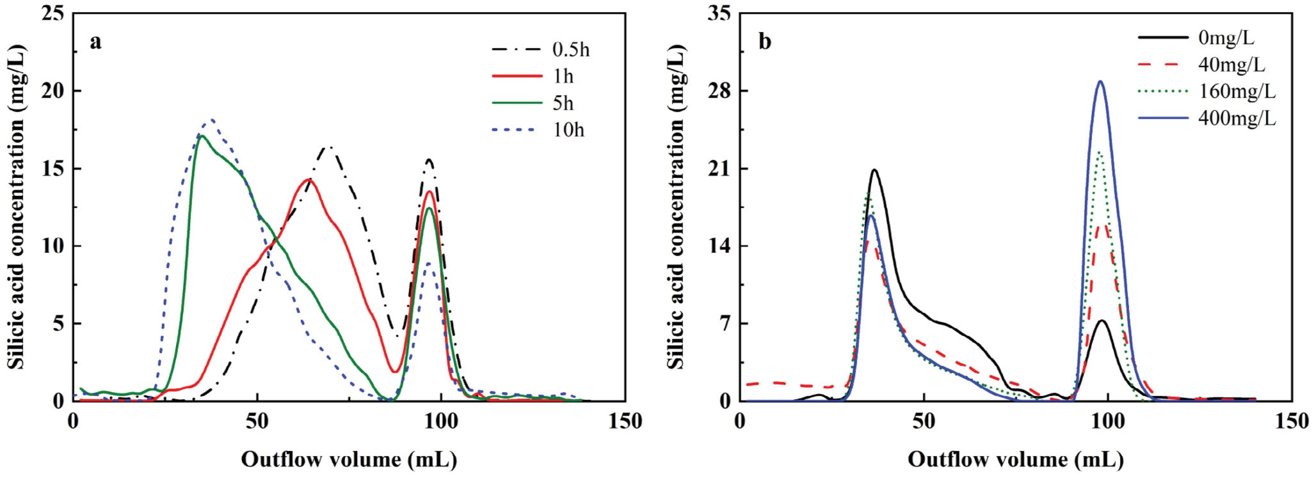

Fig. 6. Gel chromatograms for silicic acid in the absence and presence of GA after various reaction times: (a) absence of GA over time and (b) presence of GA at various concentrations.

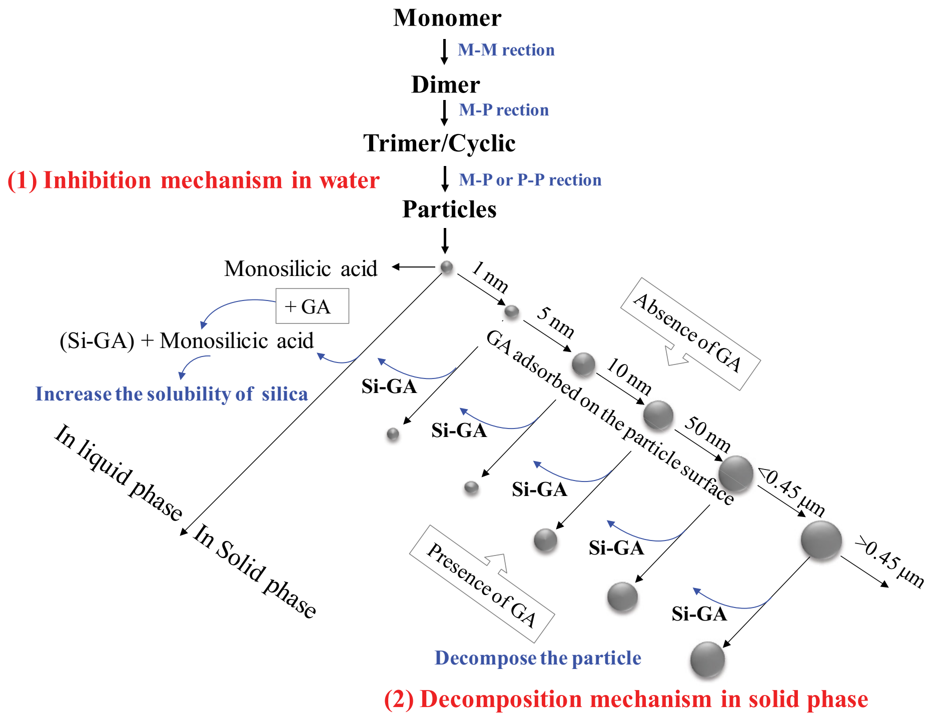

Fig. 7. Schematic diagram of inhibition and antiscaling mechanisms for GA acting on silica scale

form a soluble silicic acid complex. Increasing the solubility and reduce the probability of collision between monosilicic acid and polysilicic acid (or monosilicic acid), thus, retarding the formation of the silica scale. GA can also be adsorbed onto the surface of the silica scale to form a surface complex that decomposes the silica particles into the water as soluble silicic acid, gradually reducing the silica particle sizes. The adjacent hydroxyl and carboxyl groups in the molecular structure of GA interact with silicic acid and likewise with calcium and magnesium ions to prevent the formation of carbonate scale, showing clear potential for use in real-world water systems, especially for refractory silica scale.

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Declaration of interest statement

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Credit author statement

Shuqin Bai: Conceptualization, Writing- Original draft preparation, Methodology, Editing. Yuhan He, Ruijia Yang, Liang Yang: Data curation, Investigation. Lingzhu Cao and Yuping Ma: Software, Visualization. Jue Han: Software, Visualization, Writing- Reviewing and Editing.

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