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Study on association solubilization and inhibition of scale in recirculating cooling water system under S-HGMF

Xin Zhao, Suqin Li, Shuaishuai Han, Jianjiang Jin and Peng Zhang

ABSTRACT

The present study conducted an investigation on the effect of Superconducting-High Gradient Magnetic Field (S-HGMF) in the association solubilization of recirculating cooling water and the crystal form change of scale. The effects of magnetic flux density, flow rate and cycle-index on the solubility of scale-forming ions were investigated, and the effects of viscosity and surface tension on the molecular internal energy and order degree of circulating water were analyzed. The scale was ground and mixed with water and placed in a S-HGMF system to study the effect of S-HGMF on the crystal form change of $CaCO_3$. The experimental results showed us that S-HGMF could increase the solubility of scale-forming ions. It could enhance the interaction between water molecules by increasing viscosity and reducing surface tension, so as to improve the stability of water quality, reduce ion precipitation, and achieve the effect of scale inhibition. At the same time, it could also change the crystal structure of $CaCO_3$, promote the transformation of calcite to aragonite, and realize the purpose of scale inhibition. In a word, S-HGMF treatment can effectively solve the scaling problem of recirculating cooling water system, which provides a reference for scale inhibition of recirculating cooling water.

Key words | association solubilization, recirculating cooling water, scale-forming ions, S-HGMF, scale inhibition

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HIGHLIGHTS

- The S-HGMF is applied to scale inhibition in recirculating cooling water.
- The association solubilization effect of S-HGMF on scale-forming ions.
- S-HGMF treatment can improve the viscosity and reduce the surface tension of recirculating cooling water.
- S-HGMF treatment can transform the crystal structure of CaCO3 from calcite to aragonite.

INTRODUCTION

Recirculating cooling water contains a lot of bicarbonate, carbonate and chloride compounds, and also contains a lot of scale-forming ions, such as Ca^{2+}, Mg^{2+}, etc (Huang & Lu 2008). Since the HCO_3^- in the water are not stable, they are decomposed by heat when flowing through the heat exchange equipment and combine with the scale-forming ions to form scale (Chen *et al.* 2008). Scaling is a common problem in metallurgy, chemical industry (Ma *et al.* 2020), electric power (Okada *et al.* 2019), papermaking, cement and other industries (Charles & Douglas 1998). It is

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composed of $CaCO_3$, $Mg(OH)_2$ and $CaSO_4$, of which $CaCO_3$ is the main component and mainly in the form of calcite crystals (Li *et al.* 2019). The scale existing in the form of calcite is hard scale, which is difficult to remove when it adheres to heat exchangers, cooling towers and pipes (Zhang *et al.* 2014; Wang *et al.* 2019). $CaCO_3$ precipitation on the pipe wall will bring serious problems to the circulating cooling system (Wang *et al.* 2013). Scale not only reduce the cross-sectional area of the pipeline, the service life of equipment, the equipment stability and the heat transfer efficiency, but also increase the pipe resistance and maintenance costs. It will also affect the cooling effect and cause under scale corrosion of the system. How to achieve scale inhibition of recirculating cooling system is an urgent problem to be solved (Alimi *et al.* 2009).

At present, the commonly used scale inhibition methods in industry include chemical treatment method and physical treatment method (Zhang *et al.* 2018). The commonly chemical treatment method includ chemical softening method, ion exchange method and scale inhibitor method. Chemical treatment is widely used in recirculating cooling water systems and can effectively remove the scale. However, it has the disadvantages of high cost and secondary pollution (Apell & Boyer 2010; Mithil *et al.* 2015; Lourteau *et al.* 2019). Chemical agents will bring serious corrosion to the pipeline and shorten the service life of equipment and pipeline. Taking China as an example, a large amount of chemicals is consumed every year, which not only wastes a lot of money, but also cause serious pollution to the environment (Tian *et al.* 2008). In order to reduce the use of chemical agents, people are actively looking for other methods. In this context, physical treatment method is gradually being used to treat scale in the recirculating cooling water system (Liu *et al.* 2014; Han *et al.* 2019). Physical treatment method can change the physical or chemical properties of ions in water, and achieve the purpose of scale inhibition by changing the formation and growth process of scale. Physical treatment is easy to use, low cost and pollution-free, so it has broad application prospects and commercial market (Li *et al.* 2009; Liu *et al.* 2017; Xu *et al.* 2018). As one of the physical scale inhibition methods, magnetic treatment is gradually applied to water treatment.

Johan *et al.* (2016) installed permanent magnets in the scaling pipeline to monitor the effect of magnetic field on scaling. The results showed that scale removal effect of magnetic field on pipe wall was enhanced by 46.7%, and the removal rate was also increased to 30% with the increase of magnetic field intensity to 0.4 T. Tai *et al.* (2008) Studied the effect of permanent magnet with different magnetic induction intensity on the growth of calcite suspension in fluidized bed. The results showed that in the presence of magnetic field, the growth rate of calcite was lower than that without magnetic field, and the higher magnetic field intensity, the lower growth rate. Ammar *et al.* (2017) studied The effect of magnetic field treatment on the scaling tendency of calcium bicarbonate. The results showed that the solution of high concentration bicarbonate and calcium could inhibit scale after treated by magnetic field. Chang & Tai (2010) tested the growth of aragonite under different types and intensities of magnetic field. The aragonite seed crystals did not grow without magnetization at room temperature, but did grow under the influence of the magnetic field. The magnetic field with a higher intensity developed its effect in a shorter time. Although people have done some research on magnetic field scale inhibition, they are all carried out under the condition of low magnetic flux density. There are few studies on the treatment of recirculating cooling water with S-HGMF. No one has used S-HGMF to treat recirculating cooling water and scale. S-HGMF technology is developed on the basis of traditional ferromagnetic technology. Superconductors have no resistance at operating temperature, which shows great energy saving potential and provides a wider range of applications for this technology (Zaidi *et al.* 2014). It has been applied in the purification of magnetic metal impurities (Chen *et al.* 2012), heavy metal wastewater treatment (Kim *et al.* 2003), ecological restoration of rivers and lakes (Hu *et al.* 2014).

It has also been established that magnetic field can lead to large savings in cleaning, power consumption, time, maintenance cost (Baker & Judd 1996; Busch & Busch 1997). Li *et al.* (2012) used superconducting high gradient magnetic separation technology to treat converter turbid wastewater, It has found that this method has low equipment cost, simple operation, save water and energy, and has a great application prospect. Sohaili *et al.* (2016) discussed the influence mechanism of magnetic treatment on scale, and concluded that the technology is a simple, economic and environmental protection water treatment method, with significant scale removal efficiency. Xiao *et al.* (2020) used magnetic field to treat biological pollution in reclaimed water distribution system. Although the initial installation cost of electromagnetic treatment was high, the cost of electromagnetic treatment would be cheaper than chlorination treatment in the long run.

In this study, association solubilization in recirculating cooling water and crystal transformation of scale under the S-HGMF treatment is developed. Based on a large amount of comparative experiments, the solubility of scale-forming ions, water viscosity, surface tension, and the morphology of scale crystals was analyzed. At the same time, the mechanism of association solubilization and scale inhibition under S-HGMF would be discussed.

EXPERIMENTAL SECTION

Experimental system

Figure 1 showed the experimental flow chart of S-HGMF. The device was composed of water container, peristaltic pump, and S-HGMF in series. Superconducting magnets produced high-intensity magnetic field and the central magnetic field strength could reach 5 T (Yang *et al.* 2018).

Hardness was an important factor reflecting the solubility of scale-forming ions. When the solubility of scale-forming ions decreased, precipitation would form and the hardness would decrease. In the experiments, putted 2 L of recirculating cooling water into the S-HGMF system at first, by controlled flow rate, magnetic flux density, and cycle-index, the solubility of scale-forming ions was studied. The hardness of recirculating cooling water was measured in an conical flask and heated at temperature of 80 °C for 12 hours.

The effect of S-HGMF on the crystal transformation of scale was also investigated. In the experiments, the scale was ground and mixed with deionized water, and treated for 30 mins under the condition of magnetic flux density of 3 T. After the experiment, the scale was filtered out and the crystal structure change of scale was analyzed.

The recirculating cooling water and scale analysis

The recirculating cooling water was obtained from a steel company located in Hebei, China. The characteristics of water are listed in Table 1. It could be seen that the hardness and alkalinity of recirculating cooling water were high, and it was easy to scale.

Taking the scale for analysis, it could be seen that the scale contains a certain amount of Ca, Mg, Si, and other elements. This told us that it was a composite scale and the main elements was Ca. As shown in Table 2.

Characterization

The viscosity and surface tension changes of recirculating cooling water were measured by viscometer and surface tension meter. X-ray fluorescence (XRF) and X-ray diffraction

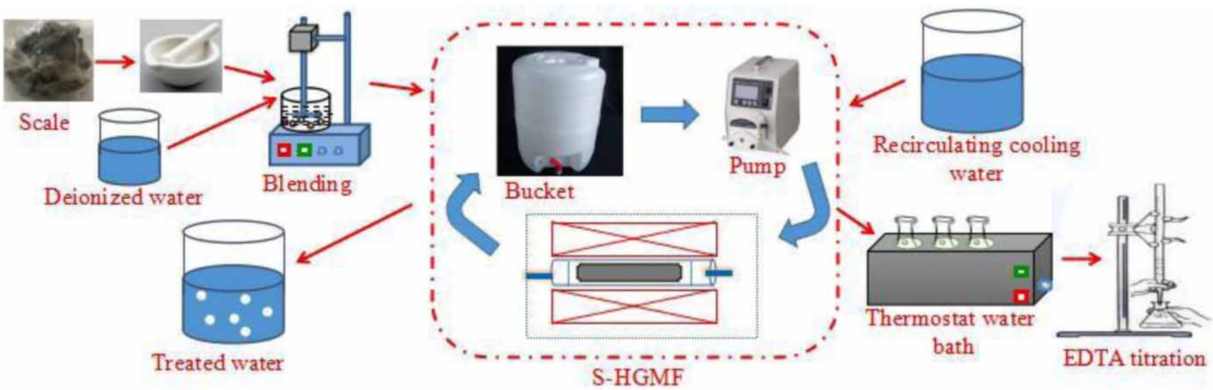

Figure 1 | Experimental flow chart of S-HGMF treatment.

Table 1 | Analysis results of recirculating cooling water

Parameters	Value
pH	8.35
Hardness (mg/L) (as $CaCO_3$)	400–600
Alkalinity (mg/L) (as $CaCO_3$)	220.76
Fe (mg/L)	0.27
SiO_2 (mg/L)	6.57
Conductivity (μS/cm)	1,852
Cl^- (mg/L)	59.87
SS (mg/L)	7.44
NH_3-N (mg/L)	3.57
COD (mg/L)	22.57

Table 2 | XRF analysis of scale from recirculating cooling water system

Element	Content (%)
Ca	46.31
O	34.40
Mg	4.94
Si	3.37
P	3.37
Zn	2.63
S	1.41
Fe	1.34
Na	0.62
Al	0.49

(XRD) analysis of scale composition and phase composition. Japan FE-JSM 6700 scanning electron microscope (SEM) was used to observe the morphology of scale crystals.

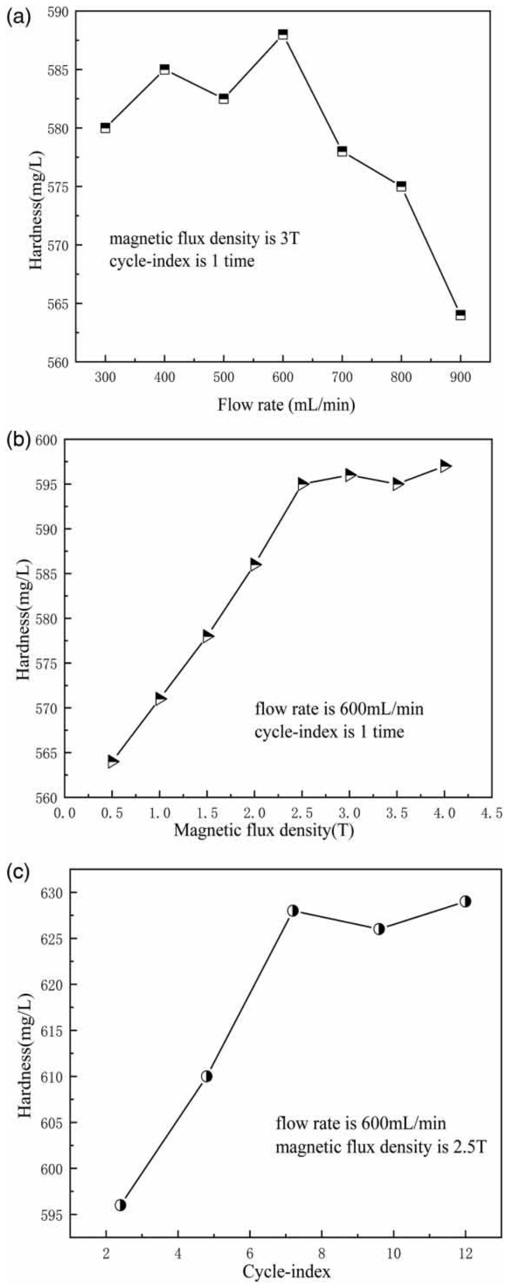

Figure 2 | The influence of (a) flow rate, (b) magnetic flux density and (c) cycle-index on association solubilization.

RESULTS AND DISCUSSION

Association solubilization under S-HGMF

Under magnetic flux density of 3.0 T, changed the flow rate, the association solubilization of scale-forming ions was discussed, the results are shown in Figure 2(a). During the flow rate from 300 mL/min to 600 mL/min, the hardness was basically stable. As the flow rate increased, the hardness gradually decreased. The reason might be that scale-forming ions in the water reacted with HCO_3^- to form precipitates under the heating condition of the water bath. When the flow rate was low, S-HGMF could have a better effect on the ions. It prevented the formation of scale, and the water hardness was higher. As the flow rate increased, the scale-forming ions stayed in the S-HGMF equipment for less time, and the hardness was gradually reduced.

Under flow rate was 600 mL/min, changed the magnetic flux density, the association solubilization of scale-forming ions was discussed, as shown in Figure 2(b). The results showed that when the magnetic flux density was low, the hardness of recirculating cooling water was low under the heating condition of the water bath. With the increased of magnetic flux density, the hardness began to increase. There were a lot of scale-forming ions in recirculating cooling water. After water bath treatment, the solubility of scale-forming ions decreased and precipitates are formed. So the hardness decreased; After S-HGMF treatment and changed the magnetic flux density, the hardness of recirculating cooling water increased gradually. It showed that S-HGMF could improve the solubility of scale-forming ions and reduced the precipitation. When the magnetic flux density was greater than 2.5 T, the hardness would not change. So the magnetic flux density of 2.5 T was the best treatment condition.

Under flow rate was 600 mL/min and magnetic flux density was 2.5 T, changed the cycle-index. The time for the water circulate once was 2.4 min, and cycle-index was 1, 2, 3, 4, and 5, respectively. Discussed the influence of cycle-index on association solubilization of scale-forming ions, as shown in Figure 2(c). It could be seen that with the cycle-index increased, the hardness increased. When the S-HGMF treatment at three cycles, the hardness was basically unchanged. The reason might be that the magnetic flux density was constant, with the cycle-index increased, the hardness of recirculating cooling water increased, so the solubility of scale-forming ions increased.

Effect of S-HGMF on internal energy of water molecule

Viscosity is an internal characteristic of fluid, which provides flow resistance. The viscosity of liquid water can reflect the strength of the interaction between water molecules. It changes significantly with temperature, but the studies have found that magnetic fields can also affect viscosity (Toledo *et al.* 2008). This study used S-HGMF to treat recirculating cooling water, analyzed the effect of S-HGMF on viscosity, and explored the effect of S-HGMF on the internal energy changes of water molecules.

According to Eyring theory, the viscosity of liquid water η (mPa·s) satisfies the following equation (Eyring 1936):

$$\eta = \frac{hN_A}{V_m} \cdot \exp\frac{E'}{RT} \tag{1}$$

where: h is Planck's constant, J·s; N_A is Avogadro's constant, 1/mol; Vm is molar volume of the liquid, mL/mol; E' is the molar activation energy, J/mol, R is the gas constant, J/(mol·K), and T is the absolute temperature, K.

Regarding the activation energy E' as the energy required for the space that can accommodate a water molecule in the recirculating cooling water, the change in the molar energy of recirculating cooling water ΔE and the change in the molar activation energy $\Delta E'$ satisfy the equation (Tabor 1991):

$$\Delta E = -\Delta E' = -(E' - E_0') \tag{2}$$

Through Equations (1) and (2), we can get

$$\frac{\Delta \mathrm{E}}{RT} = \ln\left(\frac{\eta_0}{\eta}\right) \tag{3}$$

η_0 and η are the viscosity of the water before and after treatment by S-HGMF, (mPa·s); E' and E'_0 are the molar activation energy of liquid water when the viscosity is η and η_0, J/mol. It can be known from the Equation (3) that the change in internal energy ($\Delta E/RT$) of the liquid can be obtained by measuring the viscosity.

The relationship between flow rate and viscosity was shown in Figure 3(a). When the flow rate was low, the effective magnetization time of recirculating cooling water through S-HGMF device was increased, and the viscosity increased. With flow rate increased, the viscosity was decreased. S-HGMF treatment increased the molar internal energy of the recirculating cooling water, weakened the interaction between molecules, and enhanced the interaction of scale-forming ions. Scale-forming ions accelerated the reaction to form precipitates and reduced the solubility. This theory accorded with the analysis result of Figure 2(a).

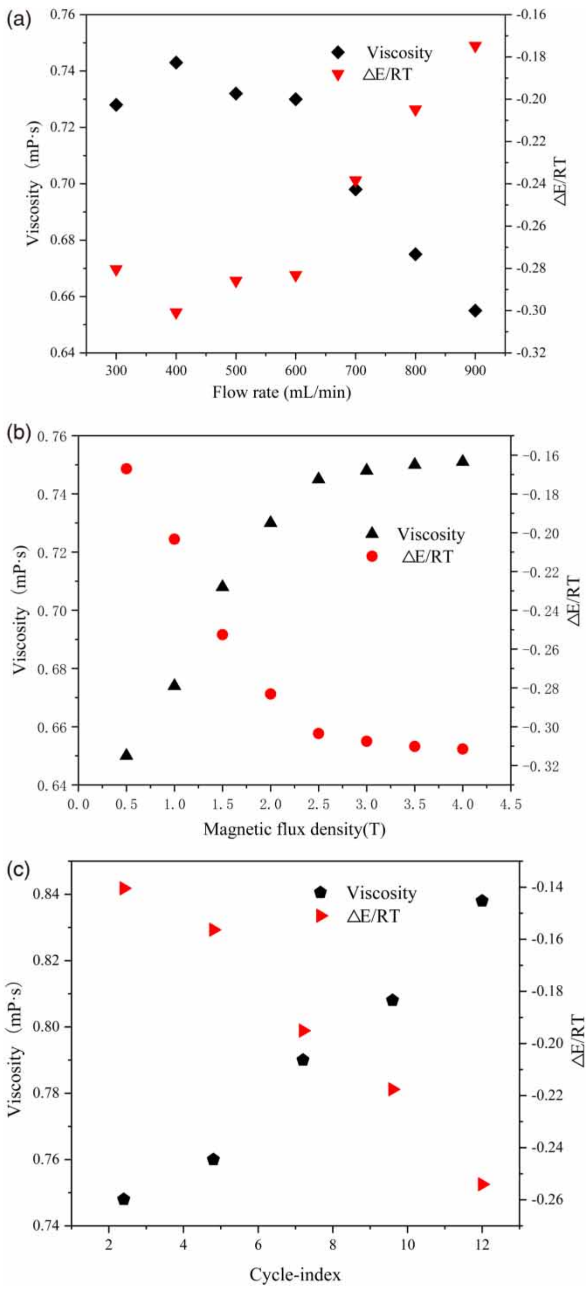

Figure 3 | The effect of (a) flow rate, (b) magnetic flux density and (c) cycle-index on viscosity.

As shown in Figure 3(b), the viscosity of recirculating cooling water increased continuously with the increased of magnetic flux density. It could be seen from Equation (3) that with the viscosity increased, the internal energy change $\Delta E/RT$ was decreased. S-HGMF treatment enhanced the interaction between water molecules and improved the stability of water quality. So the scale-forming ions needed more energy to pass through the liquid-solid interface. S-HGMF reduced the nucleation rate of scale-forming ions, and the amount of scale formation decreased, thereby achieved the effect of association solubilization. This theory accorded with the analysis result of Figure 2(b). So it was concluded that the association solubilization of scale-forming ions was related to the increase of magnetic flux density (Ghauri & Ansari 2006; Holysz *et al.* 2007).

As shown in Figure 3(c), the viscosity increased with the increased of cycle index. The results showed that with the cycle index increased, S-HGMF treatment increased the effective magnetization time of circulating cooling water, decreased the change of internal energy $\Delta E/RT$, and enhanced the stability of water quality. So the solubility of scale-forming ions increased and the hardness of recirculating cooling water increased. This conclusion was consistent with the experimental results in Figure 2(c).

Effect of S-HGMF on the order degree of water molecule

The surface tension of liquid is related to the microscopic molecular structure (Amiri & Dadkhah 2006; Lee *et al.* 2013). When the surface tension changes, the microscopic structure of the molecules in the liquid also changes, which reflects the change in the order degree of molecules inside the liquid. The effect of S-HGMF on surface tension was analyzed, and the order degree of recirculating cooling water was explored in this study.

Under certain temperature and pressure conditions, surface tension σ (mN/m) is equal to the partial differential of Gibbs free energy G(J) to surface area A(m^2) (Tabor 1991):

$$\sigma = \left(\frac{\partial G}{\partial A}\right)_{T,P} \tag{4}$$

According to the thermodynamic relationship between Gibbs free energy, enthalpy value H(J), and entropy value S(J/K), at a certain pressure P(Pa) and temperature T, the surface entropy S^A(J/(K·M^2)) satisfy the equation:

$$\left(\frac{\partial G}{\partial A}\right)_{T,P} = \left(\frac{\partial S}{\partial A}\right)_{T,P} = -S^A \tag{5}$$

In addition, the surface tension coefficient and temperature satisfy the following equation:

$$\sigma = \sigma_a \left(1 - \frac{T}{T_c}\right)^n \tag{6}$$

σ_a is the constant related to the liquid, n is the experimental factor, and Tc is the dissolution temperature. Combining Equations (4) and (5), the relationship between surface entropy S^A and surface tension can be obtained (Tabor 1991):

$$S^A = \frac{n}{T_c - T}\sigma \tag{7}$$

Let the relative change of surface entropy be $\Delta S^A/S^A{}_0 = (S^A - S^A{}_0)/S^A{}_0$. It can be concluded:

$$\frac{\Delta S^A}{S^A_0} = \frac{\sigma - \sigma_0}{\sigma_0} \tag{8}$$

S^A_0 and σ_0 are the surface entropy (J/(K·m^2)) and surface tension coefficient of untreated water, respectively. It can be seen from Equation (8) that by measuring the surface tension coefficient, the relative change in the surface entropy ($\Delta S^A/S^A{}_0$) of the recirculating cooling water can be obtained.

The flow rate was an important factor affecting the S-HGMF treatment. With the flow rate increased, the surface tension was also increased. It could be seen from Equation (8) that $\Delta S^A/S^A{}_0$ increased and the order degree of water was decreased, as shown in Figure 4(a). With increased of flow rate, the effective magnetic treatment time of recirculating cooling water was shorted and the stability of scale-forming ions decreased with the decreased of the order degree (Cai *et al.* 2009). So, it could accelerated the formation of precipitation and decreased the solubility of water. This conclusion was consistent with the experimental results of Figure 2(a).

As shown in Figure 4(b), with the magnetic flux density increased, the surface tension of recirculating cooling water was decreased and the relative change of $\Delta S^A/S^A_0$ was decreased. This result showed that S-HGMF treatment could reduce the entropy, order the molecules and increase the stability in the water (Cai *et al.* 2009). The precipitation of scale-forming ions needed more energy, so the solubility of scale-forming ions increased and the hardness tolerance of circulating water increased. This conclusion accorded with the experimental results in Figure 2(b).

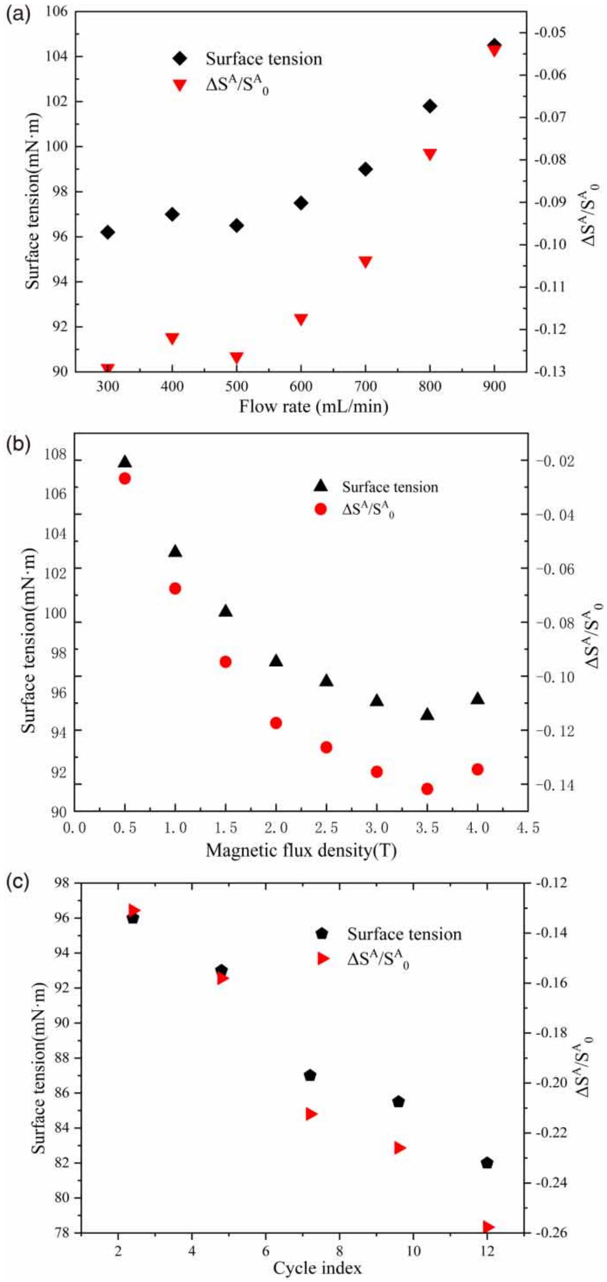

Figure 4 | The effect of (a) flow rate, (b) magnetic flux density and (c) cycle-index on surface tension.

Cycle index could affect the change of surface tension. The change of surface tension and $\Delta S^A/S^A{}_0$ decreased rapidly as the cycle index increased, as shown in Figure 4(a). With the cycle index increased, the effective treatment time of S-HGMF increased and made scale-forming ions magnetized. S-HGMF treatment made the $\Delta S^A/S^A{}_0$ decreased and the order degree increased. The chemical reaction of scale-forming ions required more energy to form precipitates and improved the solubility. This conclusion was consistent with the experimental results in Figure 2(c).

Effect of S-HGMF on scale crystal

Scale of $CaCO_3$ is an important factor affecting the recirculating cooling water system (Ammar *et al.* 2017). It is a polycrystalline crystal with many crystal types such as calcite, aragonite and vaterite. Many studies showed us that the $CaCO_3$ scale formed on the pipe wall exists as the most stable calcite crystal generally (Sohaili *et al.* 2016). In this study, the scale was treated by S-HGMF, and the effect of S-HGMF on the crystal form of scale was discussed.

XRD analysis was performed on the scale, as shown in Figure 5. The scale was mainly composed of calcite, anhydrite and quartz, in which calcite accounted for 94.96%. As shown in Figure 5(a), calcite was the main component of scale. Figure 5(b) was the XRD diffraction pattern of scale after S-HGMF treatment. It could be seen that aragonite was formed in addition to calcite, anhydrite and quartz. The content of calcite decreased to 74.78% and the proportion of aragonite was 19.43%. Calcite was the most stable crystal structure form $CaCO_3$. In XRD analysis, a part of calcite was transformed into aragonite after S-HGMF treatment, which indicated that S-HGMF treatment could change the crystal form of calcium carbonate.

In order to analyzed the morphological changes of $CaCO_3$, SEM was used to observe the morphology of scale, as shown in Figure 6. It could be seen that $CaCO_3$ was mainly composed of calcite, as shown in Figure 6(a). Under the action of S-HGMF, calcite transformed into aragonite with low crystal hardness and loose structure, as shown in Figure 6(b). The scale in the form of calcite with a hexagonal shape prior to treatment changed to aragonite after the magnetic treatment. Calcite and aragonite were two common natural forms of $CaCO_3$. Calcite was usually associated with hard scale formation, but aragonite was not easy to form hard scale.

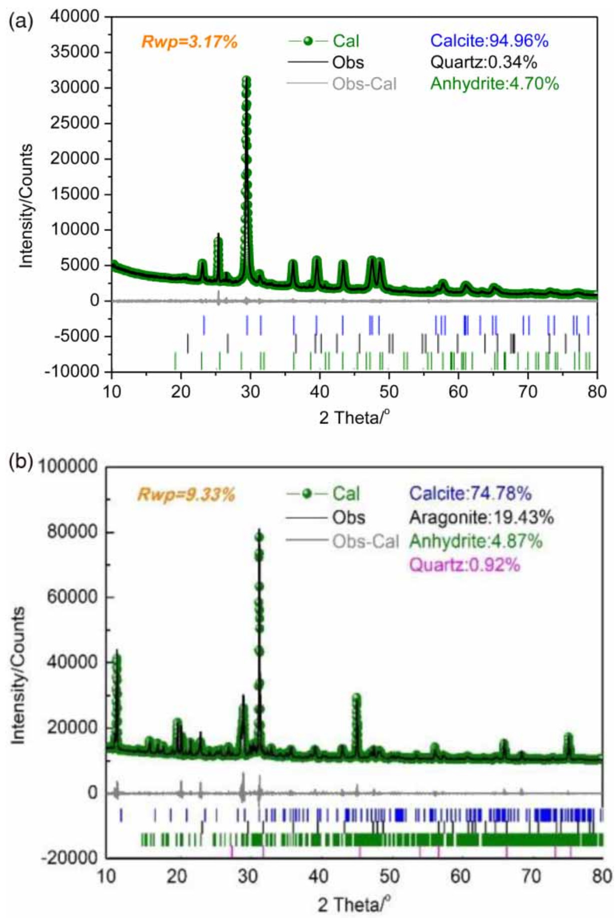

Figure 5 | XRD analysis of scale in untreated (a) and treated (b) by S-HGMF.

According to the analysis of Figure 3, S-HGMS could reduce the internal energy and resulted in lower molecular energy than that of calcite energy. Aragonite belonged to metastable structure, and its molecular energy was lower than calcite. Therefore, when the internal energy was small, it could promote the transformation of calcite to aragonite. In other words, the lower of molecular energy, the easier it was to produce aragonite. Under the action of S-HGMF, the scale was transformed from calcite to aragonite.

CONCLUSION

The present study indicates that S-HGMF technology is a good method for recirculating cooling water treatment,

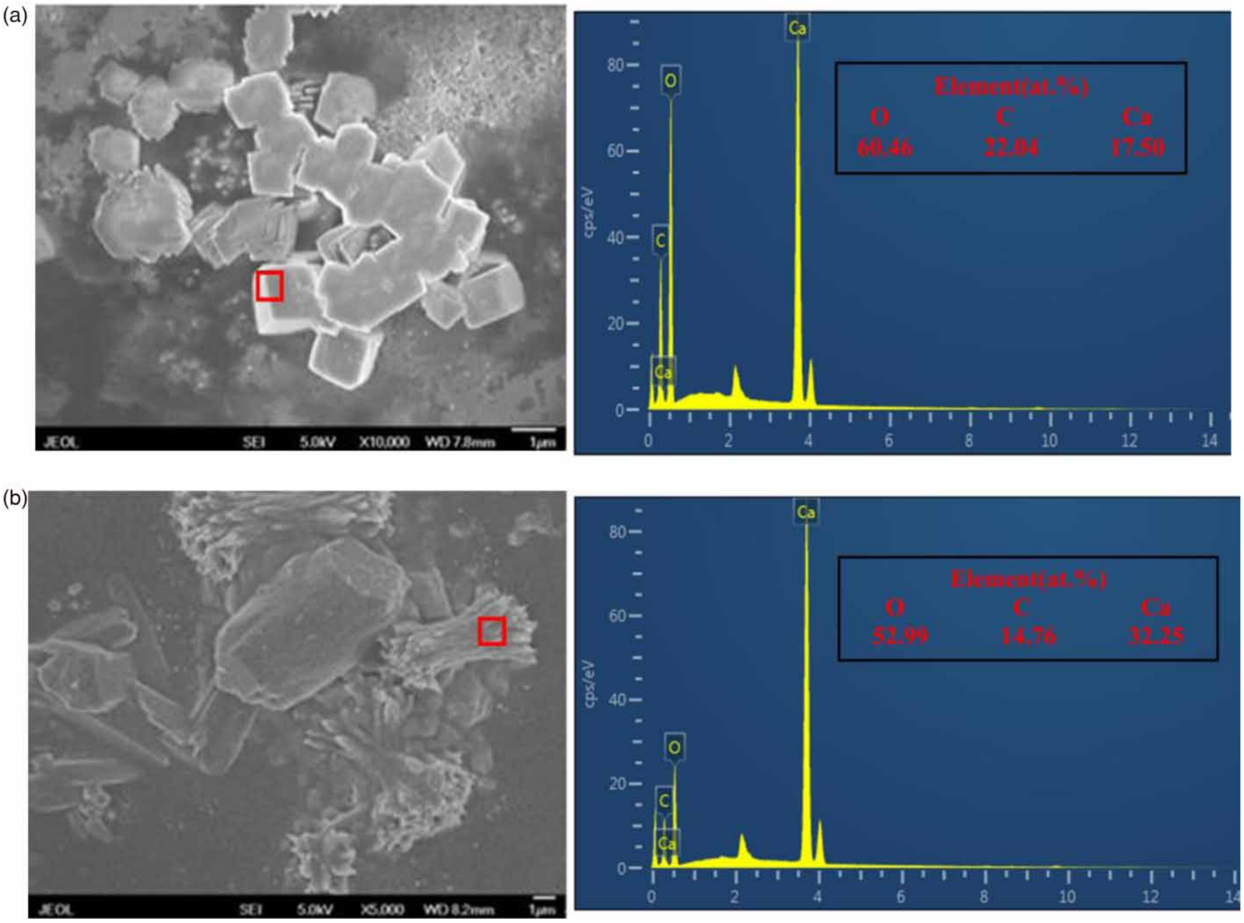

Figure 6 | SEM analysis of scale in untreated (a) and Treated (b) by S-HGMF.

and has a good effect in terms of scale inhibition. After S-HGMF treatment, reduce the internal energy of recirculating cooling water, increase the order degree of water molecules, stabilize the water quality, and reduce the scale-forming ions precipitation. It can improve the association solubilization of scale-forming ions and prevent the formation of scale. In addition, S-HGMF treatment promote the conversion of scale from calcite to aragonite.

In this study, S-HGMF treatment can reduce the precipitation of scale-forming ions, promote the change of scale structure, and prevent pipe wall scaling. It can reduce the use of chemicals and reduce the high cost of pipelines damaged by scale deposits. S-HGMF technology had the advantages of low investment, energy saving, low operating cost, convenient operation, automatic control, etc. It provides a new idea for the inhibition of scale.

CRediT authorship contribution statement

Xin Zhao: Conceptualization, Methodology, Formal analysis, Investigation, Writing-original draft, Visualization. Suqin Li: To organize and guide the formulation of the experimental scheme, experiment and result analysis and discussion; to guide the writing of manuscript and provide experimental conditions and financial support. Shuaishuai Han: Supervision, Experiment, Writing-review & editing. Jianjiang Jin: Supervision, Writing-review & editing, Peng Zhang: Supervision, Writing-review & editing.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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