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

The present study conducted an investigation on the effect of a superconducting high gradient magnetic field (S-HGMF) on 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 the circulating water were analyzed. The scale was ground and mixed with water and placed in an S-HGMF system to study the effect of S-HGMF on the crystal form change of CaCO₃. 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 CaCO₃, 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 a 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, scale inhibition, S-HGMF

HIGHLIGHTS

- 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 Ca²⁺, Mg²⁺, etc. (Huang & Lu 2008). Since the HCO₃⁻ 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), and electric power (Okada et al. 2019), papermaking, cement and other industries (Ascolese & Bain 1998).
It is composed of CaCO₃, Mg(OH)₂ and CaSO₄, of which CaCO₃ 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). CaCO₃ precipitation on the pipe wall will bring serious problems to the circulating cooling system (Wang et al. 2013). Scale not only reduces the cross-sectional area of the pipeline, the service life of equipment, equipment stability and heat transfer efficiency, but also increases 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 in a 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 methods and physical treatment methods (Zhang et al. 2018). The common chemical treatment methods include chemical softening methods, ion exchange methods and scale inhibitor methods. 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 Kumar 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, large amounts of chemicals are consumed every year, which not only wastes a lot of money, but also causes 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 methods are gradually being used to treat scale in the recirculating cooling water system (Liu et al. 2014; Han et al. 2019). Physical treatment methods 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 a 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 being applied to water treatment.

Sohaili et al. (2016) installed permanent magnets in the scaling pipeline to monitor the effect of magnetic field on scaling. The results showed that the scale removal effect of the magnetic field on the 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 magnets with different magnetic induction intensities on the growth of calcite suspension in a fluidized bed. The results showed that in the presence of a magnetic field, the growth rate of calcite was lower than that without magnetic field, and the higher the magnetic field intensity, the lower the growth rate. Al Helal et al. (2017) studied the effect of magnetic field treatment on the scaling tendency of calcium bicarbonate. The results showed that a solution of high concentration bicarbonate and calcium could inhibit scale after treatment by a 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. A magnetic field with a higher intensity developed its effect in a shorter time. Although people have done some research on magnetic field scale inhibition, this has all been carried out under the condition of low magnetic flux density. There have been 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 has been 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. 2005), and ecological restoration of rivers and lakes (Hu et al. 2014).

It has also been established that a magnetic field can lead to large savings in cleaning, power consumption, time, and maintenance cost (Baker & Judd 1996; Busch & Busch 1997). Li et al. (2012) used superconducting high gradient magnetic separation technology to treat turbid converter wastewater. It was found that this method has low equipment cost, simple operation, saves 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 environmentally protective water treatment method, with significant scale removal efficiency. Xiao et al. (2020) used a magnetic field to treat biological pollution in a 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 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 is discussed.

**EXPERIMENTAL SECTION**

**Experimental system**

Figure 1 shows the experimental flow chart of S-HGMF. The device was composed of a water container, peristaltic pump, and S-HGMF in series. Superconducting magnets produced a 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, 2 L of recirculating cooling water was put into the S-HGMF system at first, and by controlled flow rate, magnetic flux density, and cycle-index, the solubility of scale-forming ions was studied. The hardness of the recirculating cooling water was measured in a conical flask and heated at a 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 a magnetic flux density of 3 T. After the experiment, the scale was filtered out and the crystal structure change of the 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 the water are listed in Table 1. It can be seen that the hardness and alkalinity of the recirculating cooling water were high, and it was easy to scale.

Taking the scale for analysis, it can 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 element was Ca, as shown in Table 2.

**Characterization**

The viscosity and surface tension changes of the recirculating cooling water were measured by viscometer and surface tension meter. X-ray fluorescence (XRF) and X-ray...
diffraction (XRD) were used to analyze scale composition and phase composition. A Japan FE-JSM 6700 scanning electron microscope (SEM) was used to observe the morphology of scale crystals.

**RESULTS AND DISCUSSION**

**Association solubilization under S-HGMF**

Under a magnetic flux density of 3.0 T, changing the flow rate, the association solubilization of scale-forming ions is discussed, and 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

| Parameter                          | Value   |
|-----------------------------------|---------|
| pH                                | 8.55    |
| 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 ($\mu$S/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

| 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        |

**Figure 2** The influence of (a) flow rate, (b) magnetic flux density and (c) cycle-index on association solubilization.
increased, the scale-forming ions stayed in the S-HGMF equipment for less time, and the hardness was gradually reduced.

Under a flow rate of 600 mL/min, changing the magnetic flux density, the association solubilization of scale-forming ions is 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 increase of magnetic flux density, the hardness began to increase. There were a lot of scale-forming ions in the recirculating cooling water. After water bath treatment, the solubility of the scale-forming ions decreased and precipitates were formed. So the hardness decreased. After S-HGMF treatment and changing the magnetic flux density, the hardness of the recirculating cooling water increased gradually. It showed that S-HGMF could improve the solubility of scale-forming ions and reduce 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 a flow rate of 600 mL/min and magnetic flux density of 2.5 T, the cycle-index was changed. The time for the water to circulate once was 2.4 min, and the cycle-index was 1, 2, 3, 4, and 5, respectively. The influence of cycle-index on association solubilization of scale-forming ions is discussed, as shown in Figure 2(c). It can be seen that with the cycle-index increased, the hardness increased. When the S-HGMF treatment was at three cycles, the hardness was basically unchanged. The reason might be that the magnetic flux density was constant, and with the cycle-index increased, the hardness of the recirculating cooling water increased, so the solubility of the 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 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 \(\eta\) (mPa·s) satisfies the following equation (Eyring 1936):

\[
\eta = \frac{hN_A}{V_m} \cdot \exp \frac{E'}{RT}
\]

where: \(h\) is Planck’s constant, J·s; \(N_A\) is Avogadro’s constant, 1/mol; \(V_m\) is the 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 \(\Delta E\) and the change in the molar activation energy \(\Delta E’\) satisfy the equation (Tabor 1991):

\[
\Delta E = -\Delta E’ = -(E’ - E’_0)
\]

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

\[
\frac{\Delta E}{RT} = \ln \left(\frac{\eta_0}{\eta}\right)
\]

where: \(\eta_0\) and \(\eta\) are the viscosity of the water before and after treatment by S-HGMF, mPa·s; and \(E’\) and \(E’_0\) are the molar activation energy of liquid water when the viscosity is \(\eta\) and \(\eta_0\), J/mol. It can be known from 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 is shown in Figure 3(a). When the flow rate was low, the effective magnetization time of recirculating cooling water through the 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 accords with the analysis result of Figure 2(a).
As shown in Figure 3(b), the viscosity of recirculating cooling water increased continuously with the increase of magnetic flux density. It can be seen from Equation (3) that with the viscosity increasing, the internal energy change $\Delta E/RT$ was decreasing. 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 achieving the effect of association solubilization. This theory accords 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 increase of the cycle-index. The results showed that with the cycle-index increased, S-HGMF treatment increased the effective magnetization time of the 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 is consistent with the experimental results in Figure 2(c).

**Effect of S-HGMF on the order degree of water molecules**

The surface tension of a liquid is related to its microscopic molecular structure (Amiri & Dadkhah 2006; Lee et al. 2015). 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 $\sigma$ (mN/m) is equal to the partial differential of Gibbs free energy $G$ (J) to surface area $A$ (m$^2$) (Tabor 1993):

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

(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$^2$)) satisfies the equation:

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

(5)
In addition, the surface tension coefficient and temperature satisfy the following equation:

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

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

$$S^A = \frac{n}{T_c - T} \sigma$$ \hspace{1cm} (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:

$$\frac{\Delta S^A}{S^A_0} = \frac{\sigma - \sigma_0}{\sigma_0}$$ \hspace{1cm} (8)

\(S^A_0\) and \(\sigma_0\) are the surface entropy (J/(K·m²)) and surface tension coefficient of the 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 can be seen from Equation (8) that \(\Delta S^A/S^A_0\) increased and the order degree of the water decreased, as shown in Figure 4(a). With increased flow rate, the effective magnetic treatment time of recirculating cooling water was shortened and the stability of scale-forming ions decreased with the decrease of the order degree (Cai et al. 2009). So, this could accelerate the formation of precipitation and decrease the solubility of water. This conclusion is 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 \(\Delta S^A/S^A_0\) decreased. This result showed that S-HGMF treatment could reduce the entropy, order the molecules and increase 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 accords with the experimental results in Figure 2(b).

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(c). With the cycle-index increased, the effective treatment time of S-HGMF increased and made scale-forming ions magnetized. S-HGMF treatment made the $\Delta S^a/\Delta S^p$ decrease and the order degree increase. The chemical reaction of scale-forming ions required more energy to form precipitates and improved the solubility. This conclusion is 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 (Al Helal et al. 2017). It is a polycrystalline crystal with many crystal types such as calcite, aragonite and vaterite. Many studies have shown that the CaCO$_3$ scale formed on a 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 is 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 the scale. Figure 5(b) is the XRD diffraction pattern of the scale after S-HGMF treatment. It can 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 is the most stable crystal structure form of CaCO$_3$. In XRD analysis, a part of the 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 analyze the morphological changes of the CaCO$_3$, SEM was used to observe the morphology of the scale, as shown in Figure 6. It can 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 are two common natural forms of CaCO$_3$. Calcite is usually associated with hard scale formation, but aragonite does not easily form hard scale.

According to the analysis of Figure 3, S-HGMS could reduce the internal energy and resulted in lower molecular energy than that of calcite. Aragonite has a metastable structure, and its molecular energy is lower than calcite. Therefore, when the internal energy was small, it could promote the transformation of calcite to aragonite. In other words, the lower the 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, and has a good effect in terms of scale inhibition. After
S-HGMF treatment, the internal energy of the recirculating cooling water is reduced, the order degree of the water molecules is increased, the water quality is stabilized, and scale-forming precipitation is reduced. It can improve the association solubilization of scale-forming ions and prevent the formation of scale. In addition, S-HGMF treatment promotes 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 has the advantages of low investment, saving energy, low operating cost, convenient operation, automatic control, etc. It is 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 the 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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