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Experimental and mechanism research of SO_2 removal by cast iron scraps in a magnetically fixed bed

Jiang Ju-Hui *, Li Ya-Hong, Cai Wei-Min

School of Environmental Science and Engineering, Shanghai Jiaotong University, Shanghai 200240, China

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Abstract

Sulfur dioxide can be effectively removed by cast iron scraps corrosion process in a DC magnetically fixed bed, and iron sulfate compounds are gained as an available byproduct. At approximate 50 °C, when magnetic field intensity was at 15 mT and height of scraps was about 25 mm, the SO_2 removal efficiency can be kept above 80%. As the deposited rusts accumulated, the corrosion rate and desulfurization efficiency gradually decreased. The results show SO_2 removal efficiency depends on corrosion rate, and it can be obviously enhanced by DC magnetic field. With the XRD and SEM research, it can be found that DC magnetic field cannot change the crystal structure of rusts, but can make the surface morphologies on the surface of scraps looser which means easily to be removed. Consequently, the corrosion resistance can be lessened and SO_2 removal efficiency is improved significantly.
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Keywords: Magnetic field; Desulfurization; Corrosion; Cast iron scraps

1. Introduction

Pollution due to sulfur dioxide has become a serious environmental problem. Traditional methods employing lime and limestone for gas desulfurization are facing many problems, including producing a large amount of calcium sulfate for environment and huge financial burdens for coal-based power plants. Therefore, less environmentally intrusive and more cost-effective flue gas cleaning technologies need to be developed.

The catalyst of iron ions was applied in industrial flue desulfurization, the typical process being the Chiyoda [1] and the simplified reaction can be written as:

$$SO_2 + H_2O + 1/2O_2 \xrightarrow{Fe^{3+}} SO_4{}^{2-} + 2H^+ \quad (1)$$

$$SO_4{}^{2-} + 2H^+ + CaCO_3 + H_2O \rightarrow CaSO_4{\cdot}2H_2O + CO_2 \quad (2)$$

But its high-consumed energy and production of calcium sulfate have limited its popularization. In the past decade, lots of research also shows that the catalyzed oxidation of S(IV) oxides by iron ions is one of the most important factors in acid rain formation[2,3]. And in recent years, several improved methods with iron ions as catalyst have also been proposed. Wang et al. [4] proposed using microorganism (*Thiobacillus ferrooxidans*) desulfurization with ferric ion to improve the desulfurization efficiency and decrease the operational cost. Fan et al. [5,6] and Bulter et al. [7] have conducted research on the absorption of SO_2 with a solution of $FeSO_4$ to produce polymeric ferric sulfate (PFS), an inexpensive and readily available byproduct. Similarly Zhang et al. [8] have used a $FeSO_4$ solution to remove sulfur dioxide by adding iron scraps.

Then, SO_2 absorption by iron ions is still a promising flue desulfurization method. In recent years, sulfur dioxide was recognized as the main corrosive pollutant in the atmosphere [9–11], most previous research on iron corrosion by SO_2 was conducted at certain humidity, with adequate corrosion time, and for the corrosion mechanism research [9–12]. On the other hand, the magnetic field has received attention in recent years because of its theoretical implications and potential practical applications in industry, such as in metal corrosion [13–16], scale control [17,18], enhancement of mass transfer coefficient [19,20], and electrochemical reaction [21,22], etc. In this study, a new idea is put forward for the sulfur dioxide removal by the cast

* Corresponding author.
E-mail address: jiangjuhui@sjtu.edu.cn (J.-H. Jiang).

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Nomenclature

A	sulfur dioxide removal efficiency (%)
$C_{SO_{2in}}$	the inlet sulfur dioxide concentration of the column (ppm)
$C_{SO_{2out}}$	the outlet sulfur dioxide concentration of the column (ppm)
Q_G	volumetric gas flow rate (L/min)
Q_L	volumetric liquid flow rate (L/min)
Q_L/Q_G	the ratio of volumetric liquid flow rate and gas flow rate (L/m^3)

iron scraps corrosion process in a DC magnetically fixed bed and available byproduct of iron sulfate can be obtained. Experiments were conducted to determine the effects of iron scraps on SO_2 removal efficiency in DC magnetic field, and SO_2 absorption in those processes is also discussed.

2. Experiment

2.1. Materials

All the chemicals were analytical reagent and were used without further purification. Cast iron scraps were collected from the waste material in a lathe workshop. After sieving, the size was approximately 4 mm × 4 mm × 0.8 mm. Scraps contaminated by oil were degreased in acetone with ultrasonic vibration, rinsed in ethanol and dried in ventilated cabinet for 3 h at room temperature. The chemical composition of the cast iron scraps used in this work is listed in Table 1.

2.2. Apparatus and procedure

The experimental equipment is illustrated in Fig. 1, which includes a packed bed with a DC magnetic field generator, a gas supply, gas sampling ports and a liquid supply. This fixed bed is 0.05 m in i.d. and total height is 0.5 m, adequate scraps of cast iron were filled in the middle of the bed. Magnetic field generator consists of the Helmholtz coil and direct current power. Helmholtz coil was wound closely around the bed by enamel insulated wire and height amounted to 0.16 m. Enamel insulated

Table 1
Chemical composition of cast iron scraps (wt%)

Elements	Content (%)
C	3.45
Al	0.15
Ca	0.47
Cr	0.03
Cu	0.06
Mg	0.09
Mn	0.54
S	0.07
Si	0.58
Zn	0.027
Fe	Base

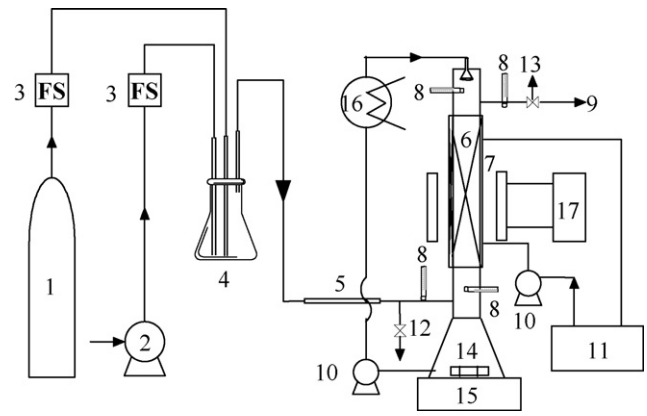

Fig. 1. Schematic diagram of the equipment used. (1) SO_2 cylinder, (2) air pump, (3) mass flow meter, (4) bufferred bottle, (5) heater, (6) packed column, (7) water jacket, (8) thermometer, (9) vent port, (10) peristaltic pump, (11) thermostatic bath, (12) gas sampling port, (13) gas sampling port, (14) holding tank, (15) magnetic force stirrer, (16) heating exchanger, and (17) magnetic field generator.

wire was provided with 3500 windings of copper wire (0.8-mm i.d). Direct current with a maximum output voltage of 230 V and a power of 300 W was available. Air was supplied by a pump, and SO_2 was added to obtain the desired concentration. Gas flow rates were measured by mass flow meters. The mixed gas was preheated by a heater.

In every experiment, 2 L de-ionized water was placed in holding tank, and solution is circularly fed to the top spraying nozzle from the holding tank through a heating exchanger with a peristaltic pump. Q_L/Q_G (the ratio of volumetric liquid flow rate and gas flow rate) was also kept 1 L/m^3, $Q_G = 10$ L/min, $C_{SO_{2in}} = 1200$ ppm (the inlet sulfur dioxide concentration of the column) and $T = 50\,^{\circ}$C (the reactor temperature) as optimized temperature, which is taken from the balance between the rate of physical absorption and chemical reaction of SO_2. All experiments reported here were carried out at atmospheric pressure and constant temperature kept by circulating temperature-controlled water through the jacket of the reactor. All experiments were reproduced at least twice, so that error was limited to ±4.5%.

2.3. Chemical analysis and rusts characterization

The concentration of Fe(II) and Fe(III) was determined by the modified 1,10-phenanthroline, in which Fe(III) was masked by ammonium fluoride. pH was measured by a CyberScan pH meter (CyberScan pH 510, Eutech Instruments Pte Ltd., Singapore). Sampling of gas at the input and output of the bed was performed intermittently by a KM900 Hand-held combustion analyzer (KM900, Kane International Ltd., UK). SO_2 absorption efficiency use the formula $A = (C_{SO_{2in}} - C_{SO_{2out}})/C_{SO_{2in}}$ to calculate.

Magnetic field intensity was determined by means of a Hall probe, and the values given here refer to the center of the bed. X-ray diffraction (Rigaku D/max 2500 pc, Cu Kα, 40 kV, 30 mA, Japan) was used to identify the corrosion products. The surface morphology was observed by scanning electron microscopy (S-2150, Hitachi Corporation). The scraps were collected by

de-ionized water washing with filter (0.22 μm) after every experiment, and were dried by vacuum freeze-dryer. The rusts were separated from iron scraps by grinding with a mortar and pestle after drying. All the samples were carefully collected, and ground to be fine powder with a mortar and pestle. Elements were analyzed by inductively coupled plasma atomic emission spectrophotometer (Iris Advantage/1000, Thermo Jarrell Ash, USA) and C/S analyzer (HCS040G, Shanghai De-Kai Instrument Co., Ltd.).

3. Results and discussion

3.1. Effect of magnetic field intensity on SO_2 removal efficiency

As can be seen from Fig. 2, two reaction stages are apparent: a rapid increase in sulfur dioxide removal efficiency (Stage I) following by a decrease in efficiency (Stage II). Sulfur dioxide can be removed to some extent by corrosion processes of cast iron scraps ($B = 0$ mT) and desulfurization efficiency was kept about 50%. Also SO_2 absorption could be enhanced effectively by DC magnetic field (from $B = 10$ mT to 20 mT). When magnetic field intensity was kept above 10 mT, SO_2 removal efficiency was enhanced at least 20%, and for 15 mT, the SO_2 removal efficiency can be kept above 80%. But, the SO_2 removal efficiency cannot be always increased with increasing magnetic field intensity. When the magnetic field intensity was above 20 mT, the SO_2 removal efficiency began to decline. In our experiments, the optimized temperature was 50 °C, and with the same flow of the circulating temperature-controlled water, the temperature of the outlet gas was 2 °C higher in the condition of $B = 20$ mT than that of $B = 15$ mT. It could be thought that decrease of SO_2 removal efficiency was caused by non-uniform temperature increase of scraps in bed at high-intensity magnetic field [19]. Generally, higher magnetic field may consume more energy and reduce the economic operation. Then in this study, the optimum DC magnetic field should be controlled at approximate 15 mT.

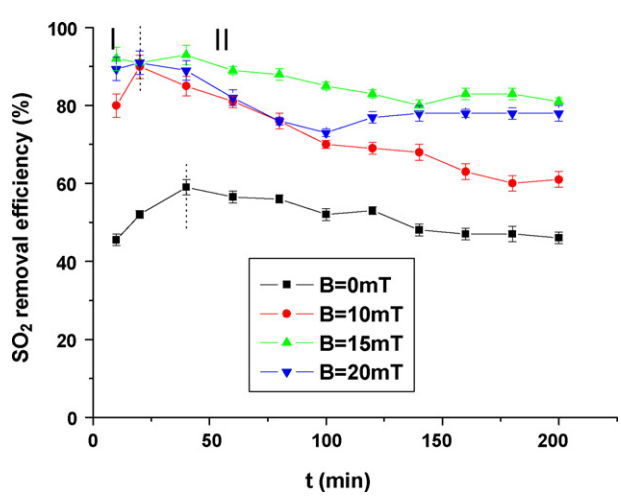

Fig. 2. SO_2 removal efficiency with different magnetic field intensity.

3.2. Corrosion processes for SO_2 absorption

In this study, SO_2 absorption can be effectively enhanced by cast iron corrosion processes in a DC magnetic field. All the processes are quite complicated, which include sulfur dioxide absorption, electrochemical corrosion, S(IV) catalyzed oxidation by Fe(III) and all the above factors could be affected by DC magnetic field.

The present work focuses on the SO_2 absorption efficiency. All those processes involved in chemical absorption into the solution include:

(i) Diffusion of the solute gas into the solution,

$$SO_2(g) \rightleftarrows SO_2(aq) \quad (3)$$

$$SO_2(aq) + H_2O \rightleftarrows H_2SO_3 \quad (4)$$

$$H_2SO_3 \rightleftarrows 2H^+ + SO_3{}^{2-} \quad (5)$$

(ii) Electrochemical corrosion (iron scraps dissolution and rusts production),

Sulfur dioxide is a medium solubility and pH is very important for its absorption. During the corrosion processes, as the spray solution was acid, the main cathodic reaction is [23]:

$$H^+ \rightarrow 1/2H_2 - e^- \quad (6)$$

Fig. 3 showed that pH can be maintained to be a relatively steady state; and in DC magnetic field, pH was higher and it can be benefited for SO_2 absorption because SO_2 is an acidic gas. In addition a high pH may decrease the corrosion rate if the hydrogen reduction is the rate-determining step.

On the other hand, anodic reactions are more complex [24]. At certain points the anodic reaction:

$$Fe \rightarrow Fe^{2+} + 2e^- \quad (7)$$

The OH^- ions react with Fe^{2+} ions to form ferrous hydroxide:

$$Fe^{2+} + 2OH^- \rightarrow Fe(OH)_2 \quad (8)$$

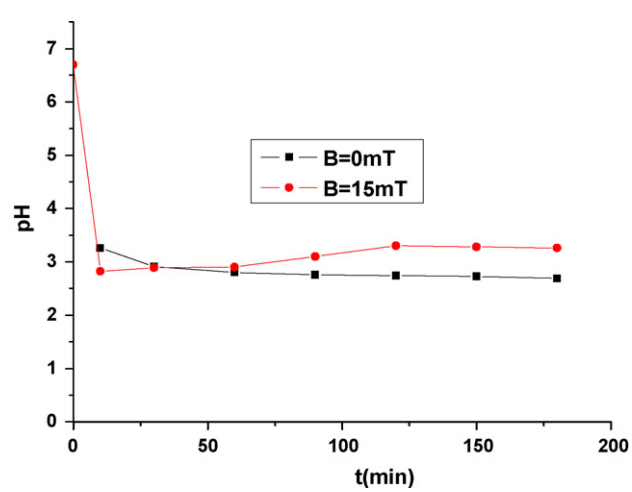

Fig. 3. Variety of pH in spray solution.

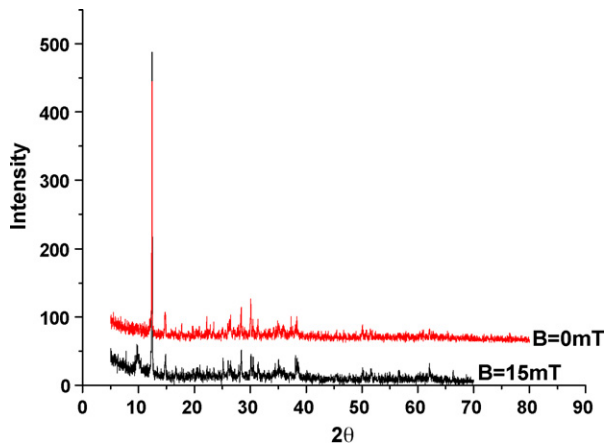

Fig. 4. X-ray diffraction patterns the rusts.

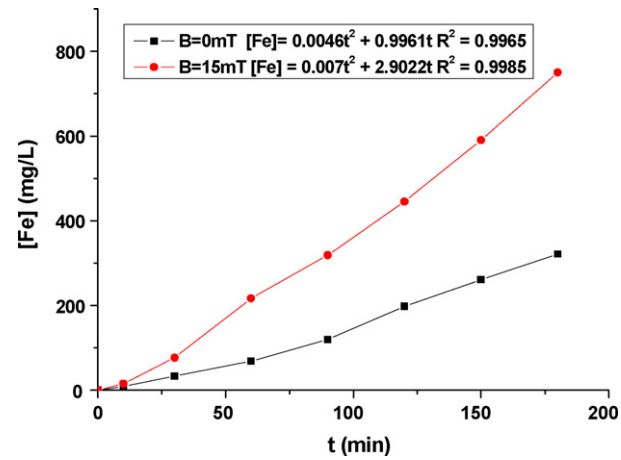

Fig. 5. Variety of [Fe] in spray solution.

This is generally oxidized to ferric hydroxide $Fe(OH)_3$, which is a simple form of rust. The final product is the familiar reddish brown rust $Fe_2O_3{\cdot}H_2O$, of which there are a number of varieties, the most common being the α form (goethite) and the γ form (lepidocrocite). In situations where the supply of oxygen is restricted, Fe_3O_4 (magnetite) or γ-Fe_2O_3 may be formed. All these corrosion products were confirmed in previous research [9–13], and it may have inhibited further corrosion. From Fig. 2, it is shown that the sulfur dioxide removal efficiency was decreased gradually in Stage II. Although the specific surface adsorption capacity for SO_2 could be increased by the rusts, the sulfur dioxide removal was evidently restrained by the final deposited rusts no matter whether having a magnetic field or not. The chemical analysis showed that rusts are difficult to dissolve in 0.1 M hydrochloric acid. It can confirm that SO_2 absorption was obviously restrained by rusts deposition. The morphologies of rusts on the cast iron scraps were investigated by XRD. As shown in Fig. 4, a strong peak appears at approximate 12.4°, which is completely different from that of previous researches in humid environment containing SO_2 [9–11,23] and cannot be identified by the XRD database (Joint Committee on Power Diffraction Standards). It could only have general information that substance containing hydroxyl might have a strong peak in this position. With the ICP and C/S analysis, the Fe and S were the major elements in rusts. Therefore, it is reasonable to assume that the rusts are the iron salt of hydroxylsulfate and hydroxylsulfite, which can restrain corrosion and decrease the SO_2 absorption efficiency.

(iii) Dissolution of iron oxides (ferric ions production).

In corrosion processes, part of rusts and iron oxides can be dissolved in acid solution and a certain amount ferric ions must be produced. Fig. 5 showed the variety of iron ions concentration during the SO_2 absorption processes, and the fitting equation of [Fe] via time can be described by: $[Fe]_{B=0\,mT} = 0.0046t^2 + 0.9961t$, $[Fe]_{B=15\,mT} = 0.007t^2 + 2.9022t$. In DC magnetic field, the increased rate of iron ions concentration was quicker than that of without field.

(iv) Chemical reaction (Fe(III)–S(IV) (catalytic oxidation reaction)).Iron ions are very important catalyst for sulfite oxidation, then it can also cause the SO_2 enhancement absorption by the Fe(III)-catalyzed autoxidation of S(IV) oxides. The simplified controlling reaction can be expressed by [25]:

$$Fe^{3+} + HSO_3^- \rightarrow FeSO_3^+ + H^+ \quad (9)$$

$$FeSO_3^+ \rightarrow Fe^{2+} + SO_3^{\bullet -} \quad (10)$$

The Fe(III)–S(IV) system is very complicated, and most of the published reaction mechanisms for homogeneous iron catalyzed autoxidation of S(IV) oxides suggest radical mechanisms [3,26,27], including a slow rate of formation of the ferric sulfite complex $FeSO_3^+$ and decomposition to produce the sulfite radical $SO_3^{\bullet -}$, subsequently a fast reaction with O_2 to form the peroxo-monosulfate radical $SO_5^{\bullet -}$. In these experiments, the sulfite oxidation was basically controlled by the Fe(III)-catalyzed autoxidation of S(IV) oxides.

According to the above analysis and two-film theory, elementary corrosion processes can be benefited for chemical reaction in liquid film, which includes: the protons were consumed and pH was kept relatively steady in processes (ii) and (iii), and Fe(III)–S(IV) catalytic oxidation reaction occurred in processes (iv). Then SO_2 absorption rate can be effectively enhanced by quick corrosion. At the same time, the deposited rusts could reduce corrosion rate and SO_2 absorption efficiency was also decreased to some extent in Stage II. In addition, temperature is very important in those processes. It is generally believed that, chemical reaction rates increase with increasing temperature; then the high temperature was benefited for corrosion processes and chemical reaction; but it was not benefited for SO_2 physical absorption; which was in agreement with the experimental results of decrease of SO_2 removal efficiency by the non-uniform temperature increase of scraps in Fig. 2.

3.3. DC magnetic field for SO_2 absorption

The curve in Fig. 2, showed that SO_2 absorption efficiency could be effectively enhanced by DC magnetic field. The mag-

netic field effects on these processes include: reaction kinetics, chemical equilibrium and corrosion processes. Magnetic fields effects on reaction kinetics and chemical equilibrium depend on the magnetic field intensity, whose order was about 1 T or above [28–31]. In our experiments, as the magnetic field intensity was below 20 mT, the magnetic field effects on SO_2 being transferred from gas to liquid and catalytic oxidation of ferric ions can be neglected. Then, the enhanced effect for SO_2 absorption should be attributed to enhanced corrosion processes with magnetic field. According to the past research, magnetic forces are the most important factor for the enhanced mass transfer by magnetic field in metal corrosion and electrochemical processes [14,15,18,29,32–35]. It is general to be accepted that two sources of magnetic forces are operative—the magnetohydrodynamic (MHD) force and the gradient magnetic force. Then a plausible assumption can be put forward: two kinds of magnetic field force exist in electrochemical corrosion processes, and they can cause a transport of all ions because of the difference in the magnetic susceptibility in the solution at the cast iron scraps surface with oriental direction, then the deposited rusts turned to be looser and corrosion rate was increased. The assumption was also confirmed by chemical analysis and the scanning electron microscope observation. Although crystal structure of rusts cannot be changed by DC magnetic field, more iron ions and higher pH can be obtained in Figs. 3 and 4, which showed that iron sulfate can be obtained more quickly and corrosion processes were enhanced remarkably. With scanning electron microscope observation, it can be found that the morphologies of rusts were looser with the DC magnetic field in Fig. 6. Then it can be reasonably assumed that looser rusts could be easily removed and it benefited for continuous corrosion and SO_2 absorption.

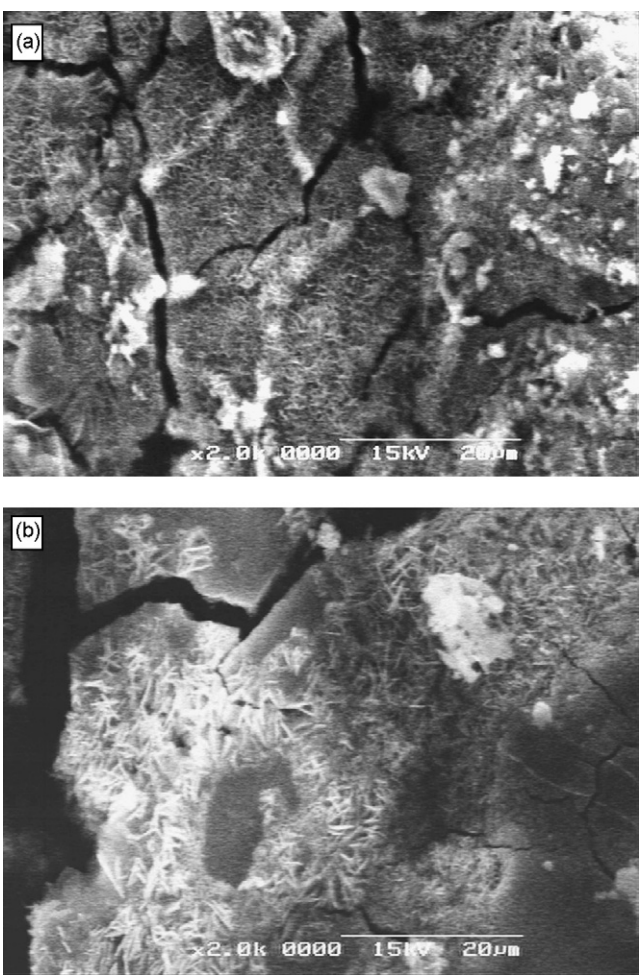

Fig. 6. Surface morphology for cast iron scraps after corrosion. (a) $B = 0$ mT and (b) $B = 15$ mT.

4. Conclusion

In this paper, SO_2 removal by corrosion process of cast scraps in a DC magnetic field has been experimentally studied.

The following may be concluded from the present work:

(1) It has been shown that SO_2 enhancement absorption can be obvious in cast iron scraps corrosion processes with the DC magnetic field and iron sulfate compounds can be obtained as an available byproduct.
(2) SO_2 removal was mainly controlled by corrosion processes, and corrosion rate can be enhanced effectively by DC magnetic field.
(3) The deposited rusts can inhibit corrosion rate and SO_2 absorption. The crystal structure of deposited rusts cannot be changed by DC magnetic field, but its surface morphologies become looser by the magnetic field forces. After that the deposited rusts can be easily removed, and rates of corrosion and SO_2 absorption were both enhanced.

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