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Effect of six kinds of scale inhibitors on calcium carbonate precipitation in high salinity wastewater at high temperatures

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ABSTRACT

Precipitation of calcium carbonate (CaCO_3) scale on heat transfer surfaces is a serious and expensive problem widely occurring in numerous industrial processes. In this study, we compared the scale inhibition effect of six kinds of commercial scale inhibitors and screened out the best one (scale inhibitor SQ-1211) to investigate its scale inhibition performance in highly saline conditions at high temperature through static scale inhibition tests. The influences of scale inhibitor dosage, temperature, heating time and pH on the inhibition efficiency of the optimal scale inhibitor were investigated. The morphologies and crystal structures of the precipitates were characterized by Scanning Electron Microscopy and X-ray Diffraction analysis. Results showed that the scale inhibition efficiency of the optimal scale inhibitor decreased with the increase of the reaction temperature. When the concentration of Ca^{2+} was 1600 mg/L, the scale inhibition rate could reach 90.7% at 80°C at pH 8. The optimal scale inhibitor could effectively retard scaling at high temperature. In the presence of the optimal scale inhibitor, the main crystal structure of CaCO_3 changed from calcite to aragonite.

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Introduction

In recent years, more attention has been paid to the reuse of refinery wastewater in the petroleum industry after advanced treatment (Liu et al., 2011a, 2011b). Compared with natural water, there are a considerable number of inorganic ions and organic substrates in refinery wastewater, which would lead to easier scale formation in circulating cooling water systems (Liu et al., 2013). Scale formation on heat transfer surfaces is a severe problem widely occurring in numerous industrial processes including batch precipitation, power generation, water transport and oil or gas production (Demadis et al., 2007; El Dahan and Hegazy, 2000; Gu et al., 2012; Suharso et al., 2011). It will reduce the efficiency of heat transfer, increase energy consumption and cause unscheduled equipment shutdowns (Yang et al., 2001).

The most common and effective scale inhibition method is to use scale inhibitors. Scale inhibition derives from complex physical processes, such as adsorption, nucleation and crystal

growth processes, rather than chemical reactions (Ketrane et al., 2009). There is little understanding of the fundamental inhibition mechanisms, especially in quantitative aspects. Generally, researchers believe that the main mechanisms of scale inhibition include: (1) the threshold effect; (2) crystal distortion effect; (3) dispersion and (4) chelation (Darton, 2000; Lisitsin et al., 2005). Among them, threshold inhibition is the most appropriate method to control scale formation (Shakkthivel and Vasudevan, 2006). In other words, even a trace amount of inhibitor can prevent a scale layer from growing or adhering to a flow surface. Such low dosages are far less than the concentration of the scaling species, so chemical additives are also called “threshold inhibitors”.

In recent years, because of the strong chelation of their functional groups with metal ions and superior dispersion characteristic of macromolecules, copolymers have been regarded as satisfactory scale inhibitors and received much attention (Wang et al., 2009). A series of studies have proved that various

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polyelectrolytes can retard crystal growth effectively (Al-Shammiri et al., 2000; Hasson et al., 1997, 1998; Smith, 1967). During crystal growth, different degrees of growth retardation for different crystal faces result in the formation of irregular crystals. The distortion of the crystal structure could increase the internal stress of crystals, which results in crystal fractures and prevents the deposition of microcrystals (Yang et al., 2001).

Polyphosphates, polyphosphonates and polycarboxylic acids are three popular copolymer scale inhibitors. Early research indicated that phosphate-containing additives had been adopted in over half of the drinking water treatment utilities in the United States (Casale, 2001). The phosphate-containing additives can sequester calcium and inhibit CaCO₃ scale precipitation even under the condition of calcite oversaturation (Marshall and Greaves, 1988). Polycarboxylic acids are another kind of copolymer scale inhibitor with cyclic or linear structure that can effectively inhibit CaCO₃ scale formation (Reddy and Hoch, 2001). The functional groups of polycarboxylic acids can chelate with Ca²⁺ ions to inhibit the formation of CaCO₃ crystals and distort the crystal lattice to generate irregular crystals.

Though in recent decades many scientists have been studying scale inhibition, few have focused on scale inhibition in high salinity and oily wastewater at high temperature. In this research, we compared the inhibition effect of six kinds of commercial scale inhibitors and screened out the optimal one to investigate its scale inhibition performance on CaCO₃ scale using the static scale inhibition method. Compared with other scale inhibition methods, the static scale inhibition method is widely used because it requires simpler devices and shorter testing time than other methods. The influences of scale inhibitor dosage, reaction temperature, heating time and pH on scale inhibition efficiency of CaCO₃ precipitation were investigated. The morphology and crystal form of CaCO₃ scale were characterized by scanning electronic microscopy (SEM) and X-ray powder diffraction (XRD) analysis, respectively.

1. Experimental

1.1. Reagents and instruments

Potassium sulfate (K₂SO₄), magnesium chloride (MgCl₂), ethylenediamine tetraacetic acid disodium salt (EDTA-2Na),

ethanol and triethanolamine were supplied by Tianjin Guangcheng Chemical Reagent Co., Ltd. (Tianjin, China). Calcium chloride anhydrous (CaCl₂), sodium chloride (NaCl) and sodium bicarbonate (NaHCO₃) were purchased from Tianjin Bodi Chemical Reagent Co., Ltd. (Tianjin, China). Eriochrome black T was obtained from Tianjin Damao Chemical Reagent Factory (Tianjin, China). Ammonium nitrate (NH₄NO₃) was purchased from Chengdu Kelong Chemical Reagent Factory (Chengdu, China). EDTA-magnesium disodium salt and ammonium chloride (NH₄Cl) were supplied by Tianjin Kermel Chemical Reagent Co., Ltd. (Tianjin, China). Ammonium hydroxide (NH₃·H₂O) was purchased from Laiyang Kangde Chemical Reagent Co., Ltd. (Laiyang, China). The six scale inhibitors are: scale inhibitor SQ1211 (Shandong TianQing Science and Technology Development Co., Ltd., China); scale inhibitor 190, 265 (Nalco Company); and LinHai-4, LinHai-1 and LinHai-3 (Shandong LinHai Science and Technology Co., Ltd., China). All the reagents above are analytical reagent grade. Deionized water was used throughout the experiment.

1.2. Evaluation of the scale inhibition efficiency of inhibitors against CaCO₃ scale

In order to screen out the best scale inhibitor among six kinds of commercial scale inhibitors against CaCO₃ scale, a preliminary screening test was carried out. The experiment was performed according to the Chinese oil and gas industry standard evaluation method (SY/T 5673-1993). The oilfield wastewater in Shengli Oil Field (China) was used. The composition and analysis of the oilfield wastewater are shown in Table 1.

Experiments were carried out in 250 mL conical flasks heated at 80°C for 24 hr. First, a certain amount of oily wastewater was added into the flasks. Then six kinds of scale inhibitors were added into the wastewater, respectively. Next, the specimens were immersed in a thermostatic water bath at 80°C for 24 hr (the level of the water bath should be higher than that of the test solutions). When the solutions naturally cooled down to room temperature, they were filtered using medium-speed quantitative filter paper. The

Table 1 – Compositions and analysis of the oilfield wastewater.

Analyte	c(1/zB ^{z-}) (mmol/L)	ρ(B) (mg/L)	Analyte	c(1/zB ^{z-}) (mmol/L)	ρ(B) (mg/L)	
Anion	F ⁻	0.000	Cation	Li ⁺	0.000	
	Cl ⁻	326.274		Na ⁺	239.990	
	NO ₂ ⁻	0.000		NH ₄ ⁺	0.985	
	Br ⁻	0.000		K ⁺	1.932	
	NO ₃ ⁻	1.197		Mg ²⁺	7.312	
	SO ₄ ²⁻	5.028		Ca ²⁺	80.109	
	OH ⁻	0.000		Sr ²⁺	0.000	
	CO ₃ ²⁻	0.000		Ba ²⁺	0.000	
	HCO ₃ ⁻	1.377				
	Amount	333.876			Amount	330.328
pH		6.5				
Degree of mineralization		19033.29	Permanent hardness		4306.51	
ρ(ΣB) (mg/L)			ρ(CaCO ₃) (mg/L)			
Total hardness		4375.43	Temporary hardness		140.13	
ρ(CaCO ₃) (mg/L)			ρ(CaCO ₃) (mg/L)			
Total alkalinity		68.92	Negative hardness		0.00	
ρ(CaCO ₃) (mg/L)			ρ(CaCO ₃) (mg/L)			

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