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Tailored surface energy of stainless steel plate coupons to reduce the adhesion of aluminium silicate deposit



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HIGHLIGHTS

• A range of Ni-P-PTFE and modified DLC coatings were applied to stainless steel plate coupons.

- These coatings significantly reduced the adhesion of aluminium silicate deposit.
- The surface energy of the coatings had significant influence on the deposit adhesion.
- The best coating reduced the deposit by 97%, compared with uncoated plate coupons.

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1. Introduction

Heat exchangers are broadly applied in chemical industries to transfer thermal energy between different fluids. However, due to the properties of the different fluids, heat exchangers are usually prone to fouling on the metal surfaces. Fouling not only reduces heat transfer performance significantly, but also causes considerable pressure drop, calling for higher pumping requirements [1]. For example, experimental results have shown that a colloidal aluminium silicate precipitate can readily form on heat exchanger plates due to the presence of the aluminium, calcium, silicon, and iron species dissolved in the plant water during the gasification of coal using a commercial Fixed-Bed Dry-Bottom (FBDB) gasification technology [2]. The plant water typically

ABSTRACT

Fouling in heat exchangers not only reduces heat transfer performance significantly, but also causes considerable pressure drop, resulting in higher pumping requirements. It would be much more desirable if surfaces which are inherently less prone towards fouling could be developed. In this paper, autocatalytic Nickel–Phosphorus–Polytetrafluoroethylene (Ni–P–PTFE) composite coatings and modified diamond-like carbon (DLC) coatings were applied to the coupons of the 316L stainless steel plates. The effects of surface energies of the coatings on the adhesion of aluminium silicate fouling were investigated and the best surface energy for which the fouling adhesion is lowest was obtained. The experimental results show that the coating with the most favourable surface energy reduced the adhesion of aluminium silicate deposit by 97%, compared with uncoated stainless steel plate coupons. The anti-fouling mechanism of the coatings was explained with the extended Deryagin, Landau, Verwey and Overbeek (DLVO) theory.

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contains 11–18 ppm Si, 2–5 ppm Al, 1–9 ppm Ca, 0.1–5 ppm Fe, 4–15 ppm Na, and 0.2–0.4% phenols with trace concentrations of K and P [2]. The formation of this gelatinous precipitate can result in severe blockages of the heat exchanger plates and will eventually require unblocking with a toxic and highly corrosive inorganic acid. The formed gelatinous precipitate or deposit on the heat exchanger plates contains 71.3% SiO₂, 26.8% Al₂O₃, 0.6% Fe₂O₃, 0.03% TiO₂, 0.01% P₂O₅, 0.12% CaO, 0.69% MgO, 0.23% Na₂O and 0.17% K₂O [2].

It would be much more desirable if surfaces with an inherently lower affinity towards fouling could be developed. Many attempts have been made to reduce fouling by coating surfaces with Polytetrafluoroethylene (PTFE) due to its non-stick properties. However, the poor thermal conductivity, poor abrasion resistance, poor adhesion to metal substrate and industrial process conditions currently inhibit their commercial use [1]. The first electroless Ni–P–PTFE composite coatings were introduced about 30 years ago [3]. The incorporation of PTFE nanoparticles into the Ni–P



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matrix can take advantage of the different properties of Ni-P alloy and PTFE. The resulting properties of electroless Ni-P-PTFE coatings, such as non-stick property, higher dry lubricity, lower friction, good wear and good corrosion resistance, have been used successfully in many industries [1]. Because the electroless Ni-P-PTFE coatings are metal-based, their thermal conductivity, anti-abrasive property, mechanical strength and adhesion strength to the substrate are superior to standard PTFE coatings [1]. Zhao et al. demonstrated that the Ni-P-PTFE coatings effectively reduce the formation of bacteria and biofilms [3–5], crystalline deposit [1,6] and food deposit [7]. Diamond-like carbon (DLC) coatings have attracted great interest due to their excellent properties such as excellent thermal conductivity similar to metals, low friction, extremely smooth surface, hardness, wear resistance and corrosion resistance [8]. The incorporation of selective elements into DLC has been shown to be an effective method to inhibit fouling formation. Zhao et al. showed that the doped DLC coatings with Si. F or N reduce bacterial attachment [9,10] and crystalline deposit [11]. Ishihara et al. [12] also demonstrated that the antibacterial performance of the pure DLC coatings was improved significantly by the incorporation of fluorine with Escherichia coli. However, to date no research has been reported for the use of Ni-P-PTFE coatings or DLC coatings to reduce aluminium silicate fouling. In this paper, autocatalytic Ni-P-PTFE composite coatings and modified diamond-like carbon (DLC) coatings were applied to the coupons of stainless steel plates and the effects of surface energies of the coatings on the adhesion of aluminium silicate fouling were investigated at the South African solvent extraction plants.

2. Experimental procedure

2.1. Ni-P-PTFE composite coatings

Ni-P-PTFE coating was prepared on 2B 316 stainless steel heat exchanger plate coupons (200 mm length \times 100 mm width \times 0.6 mm thickness) using an electroless plating technique. The stainless steel plate coupons were first cleaned with an alkaline solution at 60-80 °C for 10-20 min and then rinsed with deionised water. The composition of the alkaline solution included 25 g/l NaOH; 30 g/l Na₃PO₄; 25 g/l Na₂CO₃ and 5 g/l Na₂SiO₃. The plate coupons were dipped into a dilute hydrochloric solution (1 M) for 30 s and then rinsed with cold water and deionised water, respectively. A 60% PTFE emulsion with a particle size in the range 0.05–0.5 μ m was diluted with deionised water and stirred with a magnetic stirrer for 1 h. The solution was then filtered with a filter of pore size 0.2 µm before use. The composition of electroless Ni-P-PTFE solutions used in this investigation included 50 g/l NiSO₄·6H₂O; 60 g/l Na₃C₆H₅O₇·2H₂O; 25 g/l NaH₂PO₂·H₂O; 40 g/l NH₄CH₃COO; 4-18 ml/l PTFE (60 wt%) and 0-0.6 g/l cationic surfactant. The PTFE contents in the coatings were altered by changing the concentration of PTFE emulsion in the plating bath. The coating thickness was measured using a digital micrometer and the coating compositions were analysed with an energy dispersive X-ray microanalysis (EDX) at a beam energy of 20 keV. The surface morphology of the coatings was analysed with a scanning electron microscope (SEM). The thickness of the doped Ni-P-PTFE coating was about 10 µm, which was controlled by the deposition time. Two types of Ni-P-PTFE coatings with 4.0 wt% PTFE and 5.1 wt% PTFE were prepared.

2.2. Doped DLC coatings

The doped DLC coatings with N, Si and F were prepared on 316 stainless steel plate coupons by plasma-enhanced chemical vapour deposition technique. The stainless steel plate coupons were cleaned in an ultrasonic bath containing acetone for 10 min, rinsed

with distilled water and dried before coating. The substrates were further cleaned by Ar+ bombardment prior to deposition. Acetylene was used as the process gas for the DLC coating. The N-doped, Si-doped and F-doped DLC coatings were produced by introducing nitrogen, tetramethylsilane and tetrafluoromethane, respectively. The N, Si or F contents in the DLC coatings were altered by changing the gas flow rate of nitrogen, tetramethylsilane or tetrafluoromethane, respectively in the range between 0 sccm and 10 sccm (standard cubic centimetres per minute). The thickness of the doped DLC coatings was about 1 μ m, which was controlled by the deposition time. The N, Si or F content in the DLC coatings was analysed by EDX. The doped DLC coatings with 1.6% N, 4.1% N, 8.1% N, 7.0% Si, 5% F and 15% F were prepared. Standard DLC coatings were also prepared as a control.

2.3. Contact angle measurements

Prior to contact angle measurement, the coatings were ultrasonically cleaned in acetone, ethanol and deionised water in sequence. Contact angles were obtained using the sessile drop method with a Data physics OCA-20 contact angle analyser with an accuracy of $\pm 0.1^{\circ}$. Three test liquids were used as a probe for surface free energy calculations: distilled water, di-iodomethane and ethylene glycol. The data for surface tension components of the test liquids are given in Table 1 [13]. 6 measurements per test liquid were performed to determine the contact angles and the mean contact angles and the corresponding standard deviation were given in Table 2. The contact angles of an untreated coupon of stainless steel plate and aluminium silicate deposit were also measured. All measurements were made at 25 °C.

2.4. Surface free energy

The theory of the contact angle of pure liquids on a solid substrate was developed 200 years ago in terms of the Young equation [14]:

$$\gamma_L \cos \theta = \gamma_S - \gamma_{SL} \tag{1}$$

where γ_L is the experimentally determined surface tension of the liquid; θ is the contact angle; γ_S is the surface free energy of the solid and γ_{SL} is the solid/liquid interfacial energy. In order to obtain the solid surface energy γ_S , an estimate of γ_{SL} has to be obtained. van Oss et al. [15] developed an acid-base approach for the calculation of surface energy. The surface energy is seen as the sum of a Lifshitz-van der Waals apolar component γ_i^{LW} and a Lewis acid-base polar component γ_i^{AB} :

$$\gamma_i = \gamma_i^{LW} \gamma_i^{AB} \tag{2}$$

The acid–base polar component γ_i^{AB} can be further subdivided by using specific terms for an electron donor (γ_i^-) and an electron acceptor (γ_i^+) subcomponent:

$$\gamma_i^{AB} = 2\sqrt{\gamma_i^+ \gamma_i^-} \tag{3}$$

The solid/liquid interfacial energy is then given by:

$$\gamma_{SL} = \gamma_S + \gamma_L - 2\left(\sqrt{\gamma_S^{LW} \cdot \gamma_L^{LW}} + \sqrt{\gamma_S^+ \cdot \gamma_L^-} + \sqrt{\gamma_S^- \cdot \gamma_L^+}\right)$$
(4)

 Table 1

 Test liquids and their surface tension components [13].

Surface tension data (mN/m)	γ_L	γ_L^{LW}	γ_L^{AB}	γ_L^+	γ_L^-
Water (W), H_2O	72.8	21.8	51.0	25.5	25.5
Diiodomethane (D), CH_2I_2	50.8	50.8	0	0	0
Ethylene glycol (E), $C_2H_6O_2$	48.0	29.0	19.0	1.92	47.0

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