



# Adhesive behavior of a calcium carbonate particle to solid walls having different hydrophilic characteristics



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## ABSTRACT

In order to investigate a fouling mechanism, adhesion behavior of a calcium carbonate particle onto various walls having different hydrophilic/hydrophobic characteristics have been studied. First, adhesion force was measured by atomic force microscope, which decreases with hydrophilicity of walls. Second, fouling phenomena on each plate was measured over time. In the case of the hydrophobic surface, bare copper plate, adhesive calcium carbonate crystal was increased, and the amounts reached the constant values. On the other hand, in the case of the hydrophilic surface, glass plate and hydrophilic coated copper, calcium carbonate crystal was adhered for several hours, then the crystal was removed from the plates. Crystal types of calcium carbonate on each plate were also analyzed by a X-ray diffractometer. The crystal type was varied due to the hydrophilic/hydrophobic characteristics of the plates. From the results, it was expected that the hydrophilic treatments to wall surfaces in heat exchanger might be useful to control fouling phenomena.

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## 1. Introduction

Heat exchangers are absolutely necessary in industries, not only to keep systems safe, but also to save energy. For example, latent heat transportation is a promising technique to achieve more efficient district heating and cooling systems [1–3]. However, currently heat exchangers suffer from fouling problems. Fouling is the accumulation of undesirable deposits made of insoluble crystals on industrial equipment surfaces. These deposits, or scales, act as insulating material, and therefore the heat transfer coefficients are decreased, leading to a reduction in the efficiency of the heat exchangers. In addition, since the buildup of deposits reduces the flow rate and induces a large pressure drop in the system, the pumping power is increased. Such reductions in the efficiency of these types of systems have a significant negative effect on countries' Gross National Product (GNP) [4].

Calcium carbonate ( $\text{CaCO}_3$ ) scaling is one of the major poorly soluble deposits found on heat exchangers. While the chemical reaction to produce  $\text{CaCO}_3$  in water has already investigated [5], mechanisms of deposition on the walls of heat exchangers in flow systems are not as yet completely understood. Fouling rate is generally described as the amount of difference between rates of depo-

sition and removal [4–7]. The effect of fouling does not appear for some time after the installation or renewal of a heat exchanger. This period is called induction time, and the duration depends on system conditions [4,6,7]. In addition, since a little amount of deposit can enhance turbulence in the flow, the initial deposit built-up can increase the heat transfer rather than decrease it [4]. After the induction period, fouling resistance in heat exchangers increases.

In order to understand the fouling mechanisms and to limit fouling, many studies have been conducted [8,9]. One of a main focus of the fouling studies is the relationship between the surface of the wall and  $\text{CaCO}_3$  [10–14]. The effects of surface roughness on the fouling phenomena have been studied in experiments since the early 60s. Many researchers say that rough surfaces provide more sites for nucleation than smooth surfaces. The fouling layer makes new nucleation sites that even enhance fouling [4,10]. Therefore, crystallization fouling can easily occur on a rough surface after a short induction period. In addition, tensile stresses required to break away the fouling layer from rough surfaces are much higher than for smooth surfaces [4,10].

While many studies have focused on the surface roughness, the fouling mechanism may not be so simple. Polished smooth metal or smooth coatings are sometimes effective, however not in all reducing fouling cases [13,15–18]. In a recent study, three types of surfaces, untreated, polished and  $\text{TiO}_2$  coated surfaces were

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## Nomenclature

$b$	radius of circle in contact area between particle and wall (m)	$r_p$	radius of a particle (m)
$E_p$	Young's modulus of particle (Pa)	$\delta_p$	strain parameter of a particle ( $\text{m}^2 \text{N}^{-1}$ )
$E_w$	Young's modulus of wall (Pa)	$\delta_w$	strain parameter of a solid wall ( $\text{m}^2 \text{N}^{-1}$ )
$P$	compression force (N)	$\nu_p$	Poisson's ratio of particle (-)
		$\nu_w$	Poisson's ratio of wall (-)

tested and they were all copper-based surfaces. While the surface roughness of polished and  $\text{TiO}_2$  coated surfaces were almost the same, the amount of fouling material on the polished surfaces was the same as that found on the rough untreated surface [15]. In order to explain this result, the surface free energy was calculated from the measurement data of contact angles for each copper-based plate. The surface free energy of the  $\text{TiO}_2$  coated surface was lower than that of either the untreated or the polished plates, therefore the decrease of fouling materials on surfaces can be partly explained by low surface energy. However, in the case of  $\text{TiO}_2$  coating, the thickness of the coating also affects the fouling phenomena while the thickness doesn't change the surface free energy. Therefore, neither surface roughness, nor surface free energy, can explain the fouling phenomena completely [13]. In terms of crystal growth, a recent study looked at the growth of  $\text{CaCO}_3$  on hydrophilic and hydrophobic surfaces [19]. The study suggested that hydrophobic surfaces tended to increase crystal growth, whereas on hydrophilic surfaces, water molecules preferentially approach the surface, which reduces crystal growth [19]. Such microscopic phenomena can also affect fouling.

Atomic force microscopy (AFM) is useful to measure the adhesion force between fouling materials and surfaces directly. Experimental techniques to measure the force between particles and surface by AFM were proposed in colloidal chemistry [20,21]. In AFM, a colloid particle probe is attached to a cantilever, and the cantilever is used to approach the surface. With this technique, the repulsive force, or adhesion force between two surfaces can be detected. These forces are affected by concentrations of ions [14]. This AFM technique has applied to study surface fouling [22]. A  $\text{CaCO}_3$  particle was attached to a cantilever that approached steel surfaces of different roughness. This experiment showed that adhesion force was not linear to the surface roughness of steels. Therefore, not only the surface roughness but also other effects need to be considered.

In this study, the adhesion force of  $\text{CaCO}_3$  particles to different hydrophobic/hydrophilic surfaces was directly measured by AFM. For crystals of  $\text{CaCO}_3$ , calcite and aragonite type crystals were used.  $\text{CaCO}_3$  forms three types of crystals such as calcite, aragonite and vaterite. However, vaterite type crystal is rare in nature. Therefore calcite and aragonite type crystals were used for AFM experiments. In addition, sample plates were immersed in  $\text{CaCO}_3$  solution in order to observe which types of crystals, calcite or aragonite, appear on the plate. The crystallographic structures of  $\text{CaCO}_3$  adhered to the wall surfaces were investigated using an X-ray diffractometer (Rigaku, RINT-TTR) [23,24]. With these approaches to analysis, the investigation of the fouling mechanisms of  $\text{CaCO}_3$  can be furthered.

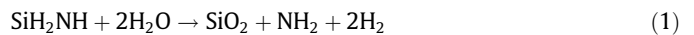
## 2. Experimental

### 2.1. Surface coating on metal plates

In this study, two types of coating, silicon dioxide (silica:  $\text{SiO}_2$ ) and silane coupling on copper plates were considered to see if hydrophilic and hydrophobic plates prevent  $\text{CaCO}_3$  fouling. Before

coating process, base copper plates were washed as follows to achieve uniform coating. First, a copper plate was immersed in acetic acid (Wako) for 3 min. Second, the plate was picked up and was washed with enough pure water. Then, ethanol (Wako) is used to dry the plate completely.

The silica ( $\text{SiO}_2$ ) coating was produced as by the follows. A small amount of the perhydropolysilazane solution (SSL-SD500, QGC-TOKYO Exousia Inc.) was dropped on a copper plate. The plate was dried for 1 h, and heated at  $500^\circ\text{C}$  for 1 h. The perhydropolysilazane reacts with the moisture in the air, and is converted to silica glass by drying at room temperature for two weeks, or by heating the copper plate at  $500^\circ\text{C}$  accelerates the following reaction, so we chose this method.

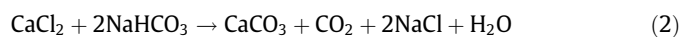


The silane coupling copper plate was produced by the following method. First, acetic acid was dissolved in equal parts of water-ethanol mixed solution. The concentration of the acetic acid in the water-ethanol solution was 2 vol%. Second, triethoxyvinylsilane (LS-815, Shinetsu Silicon Chemicals) was dissolved in the acetic acid water-ethanol solution to make the silane solution. The concentration of the triethoxyvinylsilane was 1 wt% to the weight of the plate that would be coated by the silane. Third, a copper plate was sunk in the silane solution. Then the plate was picked up and was heated to  $110^\circ\text{C}$  for 5 min. This process coupled the copper plate with silane. Ethanol was needed in silane coupling process to increase the wettability of the copper plate in solution.

The thickness of each coating was measured by a laser microscope. The average thickness of the coatings was  $0.33\ \mu\text{m}$  for silica coating, and  $0.23\ \mu\text{m}$  for silane coupling. Precise surface condition of each plate was measured with AFM (SPM-9600, Shimadzu).

### 2.2. Adhesion force measurements

The adhesion forces between calcite/aragonite to several types of plates in  $\text{CaCO}_3$  saturated solution at  $20^\circ\text{C}$  were directly measured with AFM. Since  $\text{CaCO}_3$  is insoluble materials,  $\text{CaCO}_3$  saturated solution was made by dissolving calcium chloride  $\text{CaCl}_2$  (Wako) at the concentration of 5 mM and sodium hydrogen carbonate  $\text{NaHCO}_3$  (Wako) at the concentration of 5 mM as following chemical reaction.



$\text{CaCl}_2$  reacts with half molar of  $\text{NaHCO}_3$ . Indeed, these concentration, 5 mM each, exceeded the solubility of calcium carbonate. Therefore,  $\text{CaCO}_3$  saturated solution were obtained by skimming supernatant solution. As a several plates, copper, glass,  $\text{SiO}_2$  coated copper, and silane coupled copper were used. Copper is widely used as heat-transfer materials in heat exchangers. Coated copper plates have different hydrophilic/hydrophobic characteristics. Glass plate having hydrophilic surfaces was used for the comparison. The hydrophilic characteristics of these plates were evaluated from contact angle measurement. Liquid droplets of  $\text{CaCO}_3$  saturated solution on these plates were used to check contact angles at the room temperature ( $20^\circ\text{C}$ ). A drop of  $\text{CaCO}_3$  saturated

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