



## Heterocoagulation of shale particles and bubbles in the presence of ionic surfactants



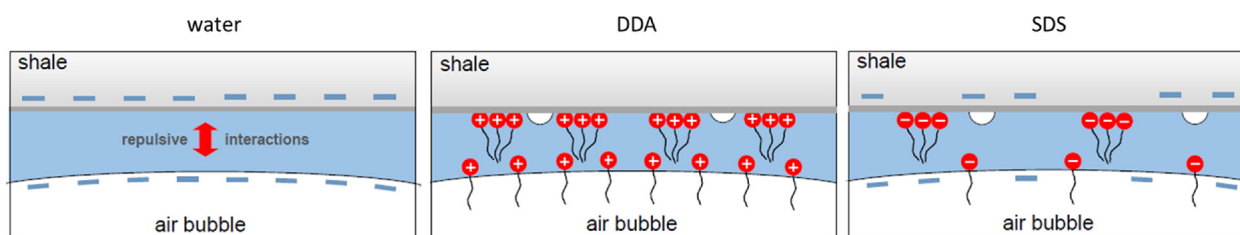
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### GRAPHICAL ABSTRACT



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### ABSTRACT

Adsorption of surfactants is one of the important feature governing interactions between particulate matters. When interactions occur between two particulates, which differ from each other, heterocoagulation takes place. This paper explains heterocoagulation of bubbles and carbonaceous metal-bearing shale particles. Heterocoagulation was experimentally studied using zeta potential, adsorption and contact angle tests. The results indicated that heterocoagulation in the presence of ionic (anionic sodium dodecyl sulfate SDS, and cationic dodecylamine hydrochloride DDA) surfactants was feasible. Based on the results, the heterocoagulation mechanism was evaluated, and thus the mechanism of shale flotation in the presence of ionic surfactants was elucidated.

### 1. Introduction

Interactions between two or more particulate matters (e.g. particles, bubbles, droplets) are of great scientific and practical interest in many areas of research and technology including separation processes. The particulate-particulate interactions determine the process kinetics and efficiency. These interactions can be controlled by surface forces leading to formation of either coagulates or heterocoagulates [1]. The term coagulation is used to describe formation of a colloidal system with the same particulates, while heterocoagulation refers to interactions of particulates, which may differ in a number of ways, e.g. type, composition, shape, size, surface charge, potential and hydrophobicity

[2]. Flotation is an example of a heterocoagulation process, where hydrophobic particles interact with bubbles in aqueous solutions.

Heterocoagulation of fine particulate matters in aqueous solutions is commonly described by the DLVO theory. The interaction between particulates (e.g. particles-bubbles) can be divided into three main types i.e. *i*) electrostatic (electrical double layer, *edl*), *ii*) dispersion (London-van der Waals), and *iii*) steric (resulted from the presence of either polymer or surfactant) [3]. According to the DLVO theory, the balance of these interactions between particulates in contact determines the coagulation process [4].

Coagulation of colloidal particulates consists of two processes, that is collision and attachment of two or more particulates, while in

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flotation detachment constitutes the third process [5]. The attachment process involves three elemental stages, i.e. *i*) thinning of a interfacial liquid film to a critical thickness, *ii*) film rupture from its critical thickness to formation of three-phase contact (TPC gas/liquid/solid), and *iii*) expansion of TPC line. The three-phase contact formation, and thus a successful particulate-particulate attachment, depends on a number of characteristics of a system including the surface charge and hydrophobicity of particulate matters, their size and roughness, temperature, water chemistry, hydrodynamics and many others.

Addition of coagulant (e.g. surfactant) destabilizes the system by changing the zeta potential and weakening repulsive hydration interactions or additional hydrophobic structural influences resulting from adsorption of a coagulant on a solid surface [6]. The extent to which the surfactant adsorbs on the solid surface is mainly determined by the value of the surface potential at certain pH. Adsorption of an ionic surfactant on the surface of particulate matter (e.g. bubble, particle, droplet) is possible due to various types of interactions of the surface and surfactant hydrophobic heads [7]. It leads to interactions between anionic surfactants with positively charged surfaces and cationic surfactants with negatively charged surfaces. The surfactant molecules adsorb with the tail facing the solution and, if a surfactant concentration is high enough, form a monolayer on the surface of particulate matter. Such modified particulates merge into aggregates by hydrophobic interactions. By considering electrostatic, hydrophobic and micellar interactions in the system adsorption of surfactants can be divided into four regions, i.e. *i*) adsorption due to electrostatic interactions between the charged sites on the mineral surface, *ii*) increased adsorption due to aggregation of hydrophobic groups of surfactants resulting in electrostatic neutralization of the surface, *iii*) adsorption due to chain-chain hydrophobic interaction, where the surface acquires the same charge as the adsorbing surfactant, and *iv*) above critical micelle coalescence *cmc*, monomer activity is constant, and thus adsorption also remains constant [8,9].

Methods to characterize heterocoagulation are diverse and include microscope, particle size distribution, zeta potential values and distribution, adsorption and others. Many researches have studied heterocoagulation in flotation [1,5,10–16], however the number of papers, which describe the interactions between bubbles and metal-bearing shale particles in the presence of ionic surfactants is limited. Therefore, the objective of the present study was to investigate and better understand the mechanism of fine shale particles and bubbles heterocoagulation in the presence of ionic surfactants through electrostatic and adsorption studies. The results were correlated with the charge of bubbles reported in literature and verified by microflotation tests.

## 2. Experimental

### 2.1. Metal-bearing shale

A geological sample of metal-bearing shale originated from the Kupferschiefer stratiform copper deposit. The shale rock samples were collected from the Polkowice-Sieroszowice mine. The chemical composition of sample was determined by X-ray fluorescence (XRF) and total organic carbon (TOC) analyses. The average percentage contents of Cu, total organic carbon, Ca, Fe, Zn, Al, Si and S were 6.0, 8.0, 11.8, 1.3, 0.3, 3.8, 10.6, 1.7, respectively. The investigated sample corresponds to the P-KS-2 shale sample described elsewhere [17]. The shale sample from Polkowice-Sieroszowice mine consisted of quartz (14%), sheet silicates (42%), carbonate minerals (33%) and copper minerals (9%) [17].

Fig. 1 shows the SEM picture of shale fraction (100–200  $\mu\text{m}$ ). Shale is polymetallic and heterogeneous substance with many different functional groups and pore structures on the surface. The determined BET surface area was equal to 4.40  $\text{m}^2/\text{g}$ .

In order to obtain the narrow size fraction of shale particles, the collected rocks were first crushed, and then sieved. Two size fractions

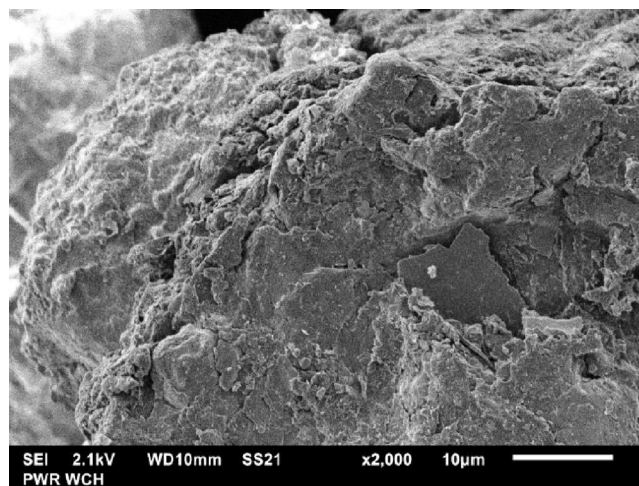


Fig. 1. SEM of shale originated from Kupferschiefer.

were used in this work *i*) 100–200  $\mu\text{m}$  in flotation tests, and *ii*) – 45  $\mu\text{m}$  in zeta potential, adsorption and contact angle measurements.

### 2.2. Surfactants

Sodium dodecyl sulfate (SDS,  $\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na}$ , MW = 223.38 g/mol), as an anionic surfactant, and dodecylamine hydrochloride (DDA,  $\text{CH}_3(\text{CH}_2)_{11}\text{NH}_2\text{HCl}$ , MW = 221.81 g/mol) as a cationic surfactant were purchased from Alfa Aesar. Dimidium bromide-disulphine blue indicator and chloroform ( $\text{CHCl}_3$ ) used in adsorption tests were purchased from Alfa Aesar. Sodium hydroxide (NaOH) and chloric acid (HCl), both purchased from POCh, were used to adjust pH. All chemicals were specified to be of the highest purity, and thus were used without further purification. High purity water with a specific conductivity of  $10^{-6}$  S/cm was used in all experiments as well as for cleaning and preparation of surfactant solutions.

### 2.3. Zeta potential measurements

Zeta potential measurements were conducted at 25  $^\circ\text{C}$  using a zeta potential analyser (Malvern Zetasizer 2000). The suspension containing 0.010 g of shale particles ( $\sim 45 \mu\text{m}$ ) in 25  $\text{cm}^3$  of aqueous solution of surfactants at the certain concentration at given pH was conditioned in a beaker for 10 min. Then, the suspension was placed in the electrophoresis cell. The value of zeta potential was determined as an average of five successive measurements.

### 2.4. Adsorption tests

Adsorption isotherms were determined by using a two-phase (water-chloroform) titration method with a dimidium bromide-disulphine blue indicator. The suspension containing 0.8 g of shale particles, with the size fraction less than 45  $\mu\text{m}$ , in 40  $\text{cm}^3$  of aqueous solution of surfactants at the given concentration was conditioned for 24 h at room temperature (25  $^\circ\text{C}$ ). Then, the solid particles were separated by filtration. The residues were dried and used in the contact angle measurements. The solution after adsorption was taken from supernatant and titrated with the surfactant solution of opposite charge (SDS for DDA isotherm and DDA for SDS isotherm). Titration was conducted to colour change of chloroform phase i.e. blue to pink for the DDA isotherm and pink to blue for the SDS isotherm. The experiments were carried out at natural pH of aqueous solutions of surfactants.

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