



Research paper

Rheological properties of montmorillonite dispersions in dilute NaCl concentration investigated by ultrasonic spinning rheometry

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ABSTRACT

Rheological changes of gelled montmorillonite dispersions with different NaCl concentrations and alkali conditions were evaluated by ultrasonic spinning rheometry. It uses velocity-profile information that is obtained in an open-cylindrical container under periodic oscillations. The measurement was conducted with a focus on the rheological behavior at a low shear rate ($O(1 \text{ s}^{-1})$), which is difficult to measure because of shear banding. The rheometry represents the coexistence of gel and sol conditions in dispersions as profiles of the phase lag of oscillations that are propagated from the cylinder wall. The critical shear rate at a yielding point and the onset of shear-thinning behavior was quantified, which has been regarded as only an apparent or speculated value by many previous researchers. Viscoelasticity from particle networks in the dispersion was observed, and the networks deform like a spring, without breaking the structure under low-shear-rate conditions.

1. Introduction

1.1. Rheology of montmorillonite dispersions

Particles of montmorillonite (Mt), which is a type of smectite, are polygonal platelets with a $O(\text{nm})$ thickness and a 0.1 to $1 \mu\text{m}$ planiform size. When Mt powder swells in ionic solvents, the dispersions provide typical rheological characteristics arising from networks of Mt particles that are termed thixotropic characteristics (Vali and Bachmann, 1988; Velde, 2013), as summarized in the review by Barnes (1997). These characteristics have been utilized in field such as agriculture, civil engineering, and nuclear engineering, as drilling muds, adsorbents, and nanocomposites. In these applications, the networks must be understood, which are structuralized passively with a time-dependence and with physical bonding forces. An anomalous viscosity or viscoelasticity of the dispersion is given by structuring networks, which are affected by particle aggregation or flocculation as a result of particle attractive or forces. Three main factors influence the structuring: Van der Waals attraction, electrostatic repulsion, and Coulomb attraction forces (Brandenburg and Lagaly, 1988; Tombácz and Szekeres, 2004).

According to Lagaly and Ziesmer (2003), the particle networks in Mt dispersions vary according to solvent pH and ionic strength as summarized in Fig. 1. The particle has electrocharacteristics of a large permanent negative face charge that arise from isomorphic substitution within the particle structure, and a pH-dependent edge charge at the

end of the structure. The negative charge is compensated by exchangeable cations. The electrostatic interaction between the particles can change easily by pH and cation concentration in the solution. These electrical characteristics promote the structuring of stable networks at microscopic scales, and therefore, the dispersions result in an anomalous viscosity or viscoelasticity in the macroscopic rheological properties (Abend and Lagaly, 2000).

Particle networks in the dispersion have various microscopic structures, which are classified into two types, namely, house-of-cards and band-type structure. The former is a stable structure with edge/face bonding, and is only formed in acidic media for edges that are positively charged, or in slightly alkaline media above the critical salt concentration (Lagaly and Ziesmer, 2003). If the concentration increases gradually beyond a critical value, the structure changes from the house-of-cards to the band-type structure (Abend and Lagaly, 2000). This occurs because the ionic strength shifts from an edge/face to a face/face network, which causes high densities of edge charges from an increase in salt concentration as shown in Fig. 1.

To understand the rheological properties of different structural networks, some factors have been investigated through experimental and theoretical research at microscopic scales (Rand et al., 1980; Bekkour et al., 2005; Goh et al., 2011). When structural transitions occur, the structure type and attractive force between each particle changes as expected from DLVO theory (Missana and Adell, 2000; Tombácz and Szekeres, 2004), which describes the forces between

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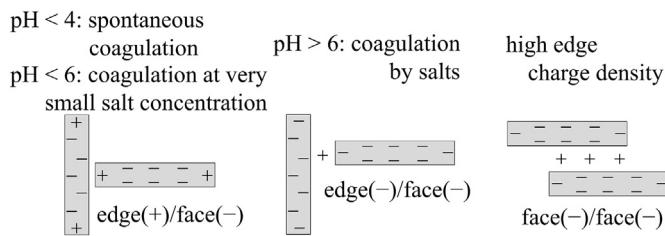


Fig. 1. The different modes of coagulation of swelling Mt particles (Lagaly and Ziesmer, 2003).

charged surfaces that interact through a liquid medium. Macroscopic responses result from these microstructures, the strength of interactions in each particle increases with respect to the salt concentration (Morvan et al., 1994). Brandenburg and Lagaly (1988) indicated that the viscosity changes significantly depending on the solvent, pH, fluid temperature, Mt powder density and sodium salt concentration, when they aimed to understand the electrical interactions between each particle.

At $pH < 6$ (isoelectric point), when particle networks are broken by a shear force, they can reconstruct instantly and spontaneously because of positive edge charges. This reconstruction is a key factor of thixotropy. In contrast, at $pH > 6$, the particles disperse homogeneously because of negative edge charges. However, salt addition allows each particle to structure networks because attractive forces exist through the cations. The thixotropy is also developed in this situation, and the thixotropic properties change with an increase in salt concentration unless the particles completely aggregate in fluid media.

A typical transition map at $pH > 6$ with changes in salt concentrations is deduced from some experimental investigations by using torque rheometry and microscopy (Brandenburg and Lagaly, 1988; Lagaly, 2006; Paineau et al., 2011; Cruz and Peng, 2016) as summarized in Fig. 2. Each structure can be broken and the particles are dispersed by shear forces, and are reconstructed by leaving them at rest. The strength of particle networks indicates that this map is divided into three phases from the rheological observations, namely, a viscous, a viscoelastic, and an elastic phases. These represent totally dispersed, flocculated, and aggregated conditions, respectively.

Many investigations have been performed to reveal the quantitative mechanical response of dispersions and to control dynamic fluid behaviors. Experimental approaches are divided into two main types; an assumption of the microstructural network from a macrorheological response (Barnes and Carnali, 1990; Nakaishi, 1997; Zhuang et al., 2017), and an assumption of the macrorheological response from microscopy (Zhang et al., 2011; Mouzon et al., 2016). Because no measurement systems exist that enable us to obtain microscopic structural deformations directly, recent research is still focused mainly on only the macromechanical response under high-shear-rate conditions by using

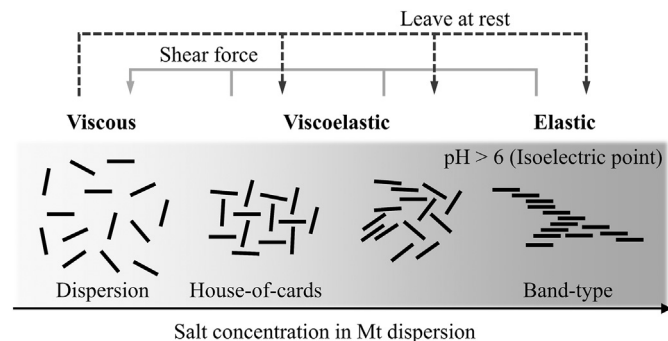


Fig. 2. Regime map of particle networks in the Mt dispersion with changes in salt concentration, shear force and leave at rest, deduced from previous studies (Brandenburg and Lagaly, 1988; Lagaly, 2006; Paineau et al., 2011; Michot et al., 2013; Cruz and Peng, 2016).

torque rheometers. Minimal research exists on the macrorheological response by using torque rheometry.

This has occurred because of limitations and problems from shear banding (Manneville, 2008; Olmsted, 2008; Divoux et al., 2016) and wall slip (Dogan et al., 2002) for applications to Bingham or Herschel–Bulkley fluids under low-shear-rate conditions, such as a co-existing gel and sol media. Few experimental investigations exist on thixotropic fluids with unsteady shearing, because torque rheometers can evaluate only macrorheological properties, including influences from the problems mentioned above. The properties are defined as an apparent viscosity or plastic viscosity, are the most significant sources of ambiguity for conventional torque rheometers, and are termed the “Couette inverse problem” (Nguyen et al., 1999; Ancey, 2005; Heirman et al., 2008).

1.2. Ultrasonic rheometry

To overcome these limitations and to reveal the rheological characteristics of dispersions with thixotropic behaviors, another rheometry approach was proposed recently in which ultrasonic waves are used (Ouriev and Windhab, 2003; Derakhshandeh et al., 2012; Gurung et al., 2016; Poelma, 2017). A rheometry using ultrasonic velocity profiling (UVP) (Takeda, 2012) was proposed; UVP can measure instantaneous velocity profiles along propagation lines of ultrasonic waves.

We proposed a rheometry using UVP for complex fluids in rotating cylindrical system without requiring an inner cylinder to measure the axial torque, termed ultrasonic spinning rheometry (USR) (Shiratori et al., 2013, 2015; Tasaka et al., 2015; Shiratori et al., 2016; Yoshida et al., 2017; Tasaka et al., 2018). This technique can be applied even for opaque liquids and for complex fluids.

With simple, open cylindrical containers, measurements of a wide range of target properties are possible. Different simple shear conditions are realized as spatial distributions by setting the oscillation frequency f and the amplitude Θ to different values. Shiratori et al. (2015) proposed a model-free USR for the quantitative evaluation of a shear-rate-dependent viscosity without using any rheological models. A supplemental axial torque measurement was used to satisfy the boundary conditions to solve the equations of motion.

Tasaka et al. (2015) used USR to evaluate the effective viscosity of a liquid with tiny bubbles as a corresponding Newtonian viscosity in a UVP measurement volume under strong oscillatory shear flows. In an analysis of this approach, the phase lag of velocity fluctuations that propagated from the oscillating cylinder wall to the inner part of the fluid is related to the local effective viscosity. Here, the viscosity is determined from local information on the phase lag, and thus the method can provide spatial viscosity distributions. In an advanced USR analysis, Yoshida et al. (2017) developed the method for general complex fluids, such as strong viscoelastic fluids and fluids with dispersed O (10 mm) solid materials. Spatial effective viscosity profiles, yield stresses, and elastic moduli of Mt dispersions were evaluated by analyzing only the instantaneous velocity profiles.

Most recently, Tasaka et al. (2018) achieved a stable evaluation of the linear viscoelasticity in bubble suspensions as complex multiphase fluids using USR. From these branches of applicability in previous reports, USR system raises the possibility of evaluating rheological properties in complex fluids in more robust methodology than conventional rheometry, such as rotational torque rheometry.

1.3. Objective

In this paper, unexamined rheological properties of Mt dispersions are evaluated by using USR. The rheological responses of the dispersion show great differences, especially under relatively low-shear-rate conditions in unsteady shear flows. USR is applicable to the existence of shear banding, which have been dealt with by using an empirical valuation because of the limitations mentioned above. USR can provide

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