



Research paper

Influence of the temperature on the rheological properties of bentonite suspensions in aqueous polymer solutions

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ARTICLE INFO

Article history:

Received 3 September 2015

Received in revised form 13 January 2016

Accepted 16 January 2016

Available online 4 February 2016

Keywords:

Bentonite

Carboxymethylcellulose

Polymer solution

Rheology

Temperature

Gel

Yield stress

Viscoelasticity

ABSTRACT

Dispersions of clay particles in aqueous polymer solutions generally exhibit complex rheological behaviours depending on the mechanical history of the material. In this paper, the effect of the temperature on the rheological properties of bentonite dispersions in aqueous carboxymethylcellulose solutions is examined. The bentonite concentration was fixed to 5% w/w while the polymer is 0.5 or 1% w/w. For stresses lower than a yield stress, the systems behaved as a gel and, above the yield stress, they flowed as liquids. The samples were characterized both in permanent and oscillatory shear flow. In the liquid like regime, the viscosity of the fluids decreased as the temperature increased. In the solid like regime, when the temperature increased, the experimental results displayed unusual behaviours of the viscosity and of the shear moduli. The increase of the chain mobility induced by the Brownian motion combined with a dissociation of the aggregates of particles could result in the formation of an elastic network with more crosslinks. This could explain why a dramatic increase of the solid-like character of the dispersions was observed.

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1. Introduction

The dispersions of clay particles in polymeric fluids (solutions or melts) are used in a wide range of applications such as formulation of drilling fluids for oil recovery (Cartalos et al., 1997) or improvement of fire resistance of fibres (Bourbigot et al., 2000; Bourbigot et al., 2002). A lot of works have been done on the rheological properties of such fluids and on clay nanocomposites (Cassagnau, 2008, Duman et al., 2012, Sun et al., 2009, Tunc and Duman, 2008, Tunc et al., 2008, Vermant et al., 2007). The specific clay atomic structure is at the origin of complex behaviours. The clay is characterized by a hierarchical structure: assemblies of layers which form particles, assemblies of particles which form aggregates and finally, assemblies of aggregates (Le Pluàrt et al., 2004) (Fig. 1A). In a review paper, (Luckham and Rossi, 1999) the role of the particle-particle and particle-polymer interactions on the colloidal and rheological properties of clay dispersions was discussed. The structure of the particle associations depends on the clay type, the layer charge, the pH and the ionic strength of the fluid and different scenarios may occur: face-to-face, face-to-edge or edge-to-edge associations (Baghdadi et al., 2005, Chang et al., 1993, Durán et al., 2000; Luckham and Rossi, 1999).

The clay dispersions in aqueous polymer solutions are generally characterized by the existence of a yield stress and of thixotropic effects,

depending on the clay and polymer concentrations. Ageing effects are of great importance in such systems (Baghdadi et al., 2005, Baghdadi et al., 2007, Coussot et al., 2002, Daga and Wagner, 2006). Such a complex behaviour is a result of the evolution of the fluid microstructure. The Herschel–Bulkley (HB) equation is often used to model the rheological behaviour of these dispersions (Besq et al., 2003, Kelessidis et al., 2011). The HB model may be modified by introducing structural parameters to take in account thixotropy (Hammadi et al., 2014). The yield stress is generally difficult to define and to measure and, during the last years, efforts have been made to clarify the situation (Mewis and Wagner, 2012, Møller et al., 2006). New approaches have been proposed using the concepts developed for soft glassy materials: the mechanical properties appear as the result of a competition between ageing and shear rejuvenation (Bonn et al., 2004). Ageing corresponds to structure rebuilt and to an increase of the viscosity vs time at low shear stresses. Shear rejuvenation corresponds to structure breakdown and therefore, to a decrease of viscosity vs. time (thixotropy). A viscosity bifurcation is observed on viscosity-time plots at given shear stresses (Møller et al., 2006). The ageing phenomena depend also on the electrostatic interactions and, in a paper devoted to aqueous laponite dispersions, Joshi et al. (2008) distinguished between the colloidal glass state (at low ionic concentration) and the gel one state (at high ionic concentration) characterized by a percolation network. A phase diagram was established for laponite/water suspensions with different amounts of salt and three different states were identified: gel, attractive glass and repulsive glass (Jabbari-Farouji et al., 2008a, 2008b).

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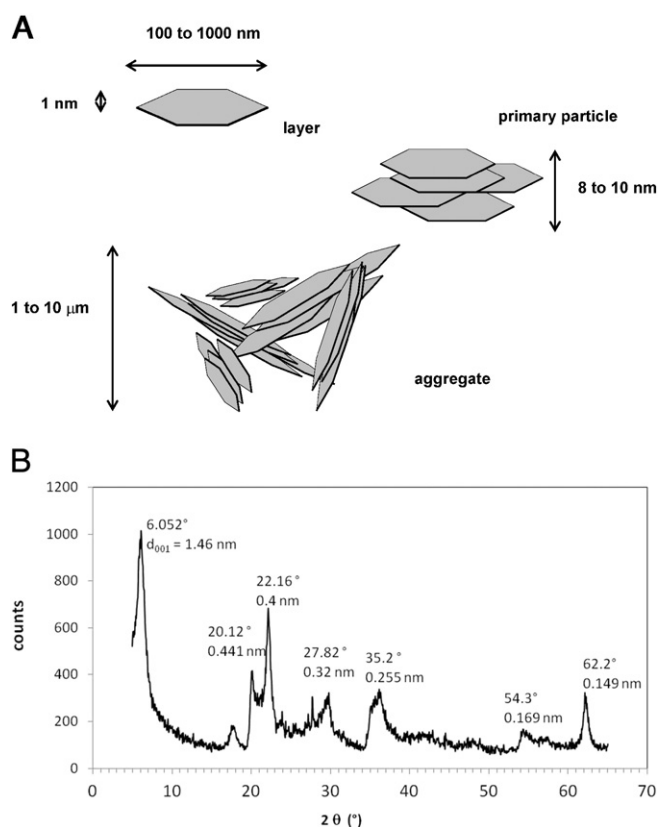


Fig. 1. A: Microstructure of clay particles; B: X ray spectrum of the bentonite sample.

Recent reviews papers concerned the different modelling approaches of such pasty systems (Cousot, 2007) and the relation between mechanical properties and micro-structure of soft matter systems (Stokes and Frith, 2008).

In polymer solution, there is a competition between clay–polymer and polymer–polymer interactions. Shearing the fluid induces particle orientation, rupture of large particle aggregates, formation of clay–polymer bonds that can be detected by flow birefringence or light scattering (De Bruyn et al., 2008, Schmidt et al., 2002).

The temperature is an important parameter, controlling the Brownian motion and the interactions between the particles and the polymer. Classical time–temperature superposition was obtained for the complex modulus G^* in the case of laponite/poly(ethylene glycol)/water dispersions (Morariu and Bercea, 2009). But, different authors report “unusual properties” for clay nanocomposites with, sometimes, sharp transitions of the viscosity or of the shear moduli. Wang et al. (2006) worked on organoclay/polybutadiene systems. Heating and cooling scans were applied to the samples and the shear viscosity was measured at different shear rates. The non-monotonous evolution of the viscosity vs. the temperature was explained by considering the balance between different effects: polymer–clay interaction which is temperature sensitive, shear induced-orientation and thermal motion. Irreversible transitions were attributed to clay exfoliation while reversible ones were attributed to orientation–desorientation of the exfoliated clays. J. Zhu et al. (2007) studied organoclay/liquid polybutadiene gels. The samples were submitted to heating and cooling scans. In some cases, a transition temperature was observed and there was a dramatic increase of the viscosity related to a structure transition from an intercalated to an exfoliated state. These non reversible transitions were attributed to the presence of strong polymer interactions.

The elastic properties of filled polymer systems can be explored through oscillatory measurements but also using creep–recovery tests. For instance, clay/poly(methyl methacrylate) nanocomposites have been studied in such a way. The increase of the elasticity of the material

with the filler content was explained assuming that the mobility of the chains attached to the particles was reduced (Muenstedt et al., 2008). The compatibility between the particles and the polymeric matrix depends on the temperature. This has important consequences on the rheological properties as has been shown on cloisite organoclay/ethylene vinyl based copolymers (Gelfer et al., 2005). Indeed, a reverse temperature dependence of the viscoelastic properties was observed: the storage modulus G' was higher at 220 °C than at 200 °C. This was explained by a decrease of the clay/matrix compatibility and a transition from an intercalated-exfoliated state to a 3D tactoid network.

In a recent paper (Ovarlez and Coussot, 2007), the effect of temperature on mechanical properties and on ageing was discussed. The authors presented different soft jamming materials: bentonite/water dispersions, TiO_2 /water dispersions and mustard. They showed that the physical age of a system is not the same at a given time if the temperature is different. Then, the definition of a reference state is quite a difficult problem.

In this paper, the focus is on the effect of temperature on the rheology of calcium bentonite suspensions in aqueous carboxymethylcellulose (CMC) solutions. Previous studies have been done on that materials (Bekkour and Kherfellah, 2002, Benchabane and Bekkour, 2006, Ben Azouz et al., 2010). At rest or at low shear stresses, they behave like elastic gels and a solid–liquid transition is observed when the shear stress increases. In this study, different independent experiments between them are presented, performed on these systems to investigate the influence of the temperature on their mechanical properties and especially on the solid–liquid transition. The samples were studied both in permanent and oscillatory shear flow.

2. Experimental

The calcium bentonite was a commercial bentonite provided by VWR Prolabo. The main components of this sample are: SiO_2 (63.9%), Al_2O_3 (14.6%), Fe_2O_3 (2.1%), MgO (2.8%), CaO (2.1%).

The bentonite concentration was fixed to 5% w/w. It was dispersed in aqueous polymer solutions. The polymer was a sodium carboxymethylcellulose (CMC) also provided by VWR Prolabo with two different grades named LV and HV (low and high viscosity) corresponding to two molecular weights (respectively 90,000 g/mol and 700,000 g/mol). In this paper, two fluids are considered: a 5% bentonite dispersion in a 1% w/w CMC LV water solution, named 1LVB5 and a 5% bentonite dispersion in a 0.5% w/w CMC HV water solution, named 0.5HVB5.

The dispersions were carefully prepared according to procedures which had been defined in previous works and after an ageing study (Bekkour and Kherfellah, 2002, Ben Azouz et al., 2010). After 24 h stirring of the CMC aqueous solution, the bentonite powder was dispersed in the polymer solution. These bentonite/CMC solution mixtures were submitted to a continuous agitation during 48 h using magnetic stirrers. After that, the samples were kept at rest during 24 h. Then, they were stirred 1 h each day during 15 days. After these different steps, the measurements were done. All the samples were kept at 20 °C in an incubator.

The X ray diffraction, Brunauer–Emmett–Teller (BET) surface area characterization and Thermogravimetric analysis (TGA) were done at the Institut de Chimie des Surfaces et Interfaces at Mulhouse on a X'PERT diffractometer from PANalytical (source: Cu; wavelength: 1.54 Å; Soller slit: 0.04 rad; receiving slit: 0.1°; step: 0.05 (°2θ); time per step: 2 s; scan: 3 à 65 (°2θ)). The X ray spectrum obtained on the bentonite sample is given on Fig. 1B. An analysis was also performed on a sample of bentonite dispersion in the CMC solution after drying and wash. The spectrum was exactly the same as for the bentonite. So, as expected, there was no exfoliation or intercalation of the macromolecular chains between the platelets, the inter lamellar distance remaining the same with and without polymer. The BET surface area obtained on a Micromeritics system was 66.43 m²/g: the bentonite was in the form of particles and not of layers. The comparison between the TGA

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