



Full Length Article

Novel parameter predicting stability of magnetic fluids for possible application in nanocomposite preparation

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ABSTRACT

Concentrated magnetite colloids in kerosene – magnetic fluids – have been synthesized. For stabilization of magnetite nanoparticles, a number of organic acids –lauric, myristic, palmitic, stearic, oleic, behenic and erucic– have been used. Particle size and structure were determined by transmission electron microscopy. Coagulation effect of 15 binary organic mixtures, containing a solvent and a precipitant, in relation to magnetic fluids has been studied in detail. Residual magnetite concentration in above precipitate liquid has been estimated photocolometrically. The sets of precipitation curves have been constructed for each stabilizer. A novel parameter for predicting of coagulation effect of precipitants has been suggested. The use of the parameter resulted in fitting of the precipitation curves into a single approximation curve. A method for quantitative determination of medium composition resulting in sedimentation or stability of magnetic fluids can be useful for manufacturing of magnetic elastomers.

1. Introduction

Concentrated colloidal solutions of ferromagnetic particles, or magnetic fluids, have evoked the interest of researchers since the 1960s, when such a fluid was first obtained and described [1]. The main property of magnetic fluids, distinguishing them from other colloids, is the ability to be attracted by a magnet at a certain concentration of the ferromagnetic phase. This property determines a wide range of application fields for these systems in science and technology [2–11].

The main preparation method for magnetic fluids is precipitation of magnetite or ferrites from aqueous solutions of metal salts. In the presence of surfactants, the particles become hydrophobic and transfer to an organic medium. Water, hydrocarbons, silicon oil and organofluorine compounds can be used as a dispersion medium, and the nanoparticles of iron, nickel, cobalt, and various ferrites can be used as a magnetic component [7,12–20].

In colloids, the intense Brownian motion, arising due to the small particle size (about 10 nm), results in sedimentation stability. However, when the particles approach each other, they begin to attract one another due to the effect of Van der Waals and magnetic forces. This way, the particle aggregates are formed and precipitated. The formation of adsorption or the double electric layers (DEL) on the particle surface prevents approaching of the particles.

In non-polar media colloids can be sterically stabilized by the

polymers soluble in an organic dispersion medium. The adsorption of the polymer on the particle surface is accompanied by the formation of macromolecular coils and loops in a solvent near the particle surface, the thickness of the stabilization layer significantly exceeding the particle size. The process of stabilizing colloids by polymers is described in detail in a monograph [21]. In many cases, the aggregative stability of colloids is achieved due to adsorption of surfactants on the particle surface. Magnetic fluids based on hydrocarbons are usually stabilized with fatty acids, in most cases by oleic acid. Thus, an adsorption layer, preventing particles from approaching to a critical distance is created.

Stabilization of colloidal magnetite in non-hydrocarbon media is mainly based on a basic rule “like dissolves in like”. Perfluorinated acids are used for stabilization in organofluorine media, surfactants with polydimethylsiloxane (PDMS) tail – for stabilization in PDMS [22]. A “universal surfactant” oleic acid can be also used for preparation of stable magnetite colloids in polydiethylsiloxane (not in PDMS) [23]. In addition, the surfactants based on polypropylene glycol (PPG) oligomer was successfully used for stabilization of magnetite in a wide range of organic solvents – from polar ones, such as ethanol and water-alcohol mixtures, excluding glycols, to completely nonpolar ones – saturated hydrocarbons and their halogen-substituted derivatives, excluding perfluorinated hydrocarbons and siloxanes [24].

The most concentrated magnetic fluids can be prepared using oleic acid as a stabilizer and kerosene as a medium. Other fatty acids are also

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Nomenclature

Components of binary mixtures

AmAc	amyl acetate
Bu-1	butanol-1
BuAc	butyl acetate
BuN	butyronitrile
Bz	benzene
BzN	benzotrile
Dig	diglyme
Diox	dioxane
EtAc	ethyl acetate

MEK	methyl ethyl ketone
Peth	petroleum ether
THF	tetrahydrofuran

Stabilizers

Er	erucic acid
Laur	lauric acid
Myr	myristic acid
Ol	oleic acid
Palm	palmitic acid
St	stearic acid

used for stabilization of this colloid in hydrocarbon media. Kerosene is a too volatile solvent for technical applications, so a solvent replacement method is widely used to overcome this disadvantage [25]. According to this technique, for coagulation of a colloid, a precipitant (acetone, ethanol, etc.) is added to MF based on kerosene, then the precipitate is separated and washed with the same precipitant. Finally, it is peptized at heating under stirring in another hydrocarbon medium, for example, in vacuum or transformer oil.

Thus, different organic solvents (acetone, ethanol, acetonitrile, etc.) have been long known to coagulate hydrocarbon-based magnetic fluids and precipitate magnetite nanoparticles.

The behavior of magnetite colloid stabilized by oleic and ricinoleic acids in some organic solvents was described in [26,27]. In [26] the colloids were supposed to be stable at dielectric permittivity of the medium $\varepsilon < 5$ and unstable at $\varepsilon > 9$. Later, in [27] it was assumed that magnetite colloid is peptized when dielectric permittivity of the medium is less than a threshold value $\varepsilon = 5$. However, this rule was refuted by the data obtained by the same authors [27]: tetrahydrofuran ($\varepsilon = 7.32$) is a good solvent for the colloids stabilized by oleic and ricinoleic acids, and *o*-dichlorobenzene ($\varepsilon = 9.8$) – for these colloids stabilized by ricinoleic acid. Experimental data described in this paper also contradict to the abovementioned works. For example, dioxane ($\varepsilon = 2$) is an effective precipitant for magnetite colloid stabilized by the whole range of fatty acids studied in present paper.

Thus, there is still no comprehensive study on the relationship between the structure and coagulation effect of precipitator.

Organic acids used for stabilization of magnetite colloids (C_{12} and longer [28]) are enough soluble in most organic solvents. In spite of this fact, a lot of organic solvents act as precipitants for all magnetite colloids, stabilized with organic acids in hydrocarbons.

Recently a lot of the studies devoted to preparation and application of core-shell type magnetic composite particles have been reported. For example, in [29–32] a number of such nanocomposites, i.e. magnetic nanoparticles with both organic and inorganic coatings, were described. These composite particles were shown to be rather efficient as catalysts for the synthesis of nitrogen-containing organic compounds. However, the information on the bulk soft magnetic materials is lacking. Looking ahead to the findings of present paper, the method described can be successfully used for preparation of bulk polymer composites filled with magnetic nanoparticles.

Dispersions of colloid magnetite in oligomers can be used for preparation of various composites [33–38], for example, magnetic elastomers, used in magnetic-coupled elements, magnetic screens, shunts, pads etc. [39]. At present nanocomposites based on magnetic filler and elastomer matrix find a wide application in science and technology. Magnetite nanoparticles have been incorporated into elastomer matrices aiming to develop new soft magnetic materials, which find applications in several fields of material science as actuators, electromagnetic wave absorbers, magnetorheological elastomers, as well as in biomedical areas, including magnetic resonance imaging, drug

delivery, hyperthermia, etc. Magnetite-based elastomer composites also have potential application as electromagnetic wave absorbers and electromagnetic interference shielding materials due to their electrical and magnetic properties [40].

Although magnetic nanoparticles are well stabilized by fatty acids in hydrocarbon media, in other solvents a problem of stabilizer-medium interaction arises. Revealing common regularities relating the structure of the medium components and stability of a resulting colloid is still very actual. A study of the particle-medium interaction would lead to the understanding of the nature and mechanism of stabilization and, hence, to the proper choice of a stabilizer-oligomer system for preparation of magnetic elastomers via curing of dispersion of stabilized nanoparticles in a functional oligomer. There are several approaches for the magnetic elastomer preparation. The most common procedure involves the melt blending of the nanoparticles with the elastomer compound in a two roll mill [41–43]. To achieve better magnetic nanoparticle dispersion, other techniques involving solution casting, dispersion in rubber latex or the polymerization/curing of liquid elastomer in the presence of the magnetite NPs have been tried. Even employing specific mixing techniques, such as sonication or high-speed mixing, the homogeneous dispersion of magnetite NPs in a polymeric matrix is not an easy task due to the van der Waals forces and magnetic dipolar interactions, which force these particles to aggregate. In this sense, coating NP surfaces with a surfactant is considered essential for improving the particle-matrix interactions and avoiding segregation of the particle phase [40].

In literature, no data were found on convenient and efficient processing of the oligomer-magnetic nanofiller mixture resulting in the composite without agglomerated particles.

A suggested method for quantitative determination of medium composition using the parameter *S* can be used for preparation of magnetic elastomers with a uniform distribution of nanoparticles. Parameter *S* of any oligomer can be calculated from the group contributions of the structural units, such as CH_2 and OH groups. This approach allows magnetic elastomers to be prepared via a simple procedure including curing of stabilized dispersion in an oligomer.

Establishment of the relationship between the chemical structure of an organic compound and its coagulation activity in respect to magnetic fluids, and, hence, predicting the behavior of a colloid in the presence of different organic compounds would complement the fundamental concepts existing in colloid chemistry.

2. Experimental

2.1. Materials

Inorganic reagents $FeCl_3 \cdot 6H_2O$, $FeSO_4 \cdot 7H_2O$ and 25% aqueous ammonia of analytically pure grade were supplied by Reachim Ltd (Russia). The following solvents of analytically and chemically pure grade were used: tetrahydrofuran (Sigma Aldrich), diglyme (Alfa-

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