Express - method for determining the quality of a magnetic fluid for operation in the working gap of a magnetic fluid seal

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Abstract. The reliability of the magnetic fluid seals operation is reduced to an assessment of the quality control of a magnetic fluid associated with its stability in the working gap of the seal in many cases. The previously developed relatively simple method for express analysis of the quality of a magnetic fluid is supplemented by studying the dependence of the dynamic characteristics of a nanofluid on the size of magnetite particles. This method can be easily implemented in an industrial enterprise using devices with a magnetic fluid.

1. Introduction

Recently, magnetic fluid seals (MFS) have found wide application for sealing systems of bearing units operating under severe operating conditions (abrasive, moisture, dust, etc.) [1]. This is due to the fact that the use of MFS instead of traditional seals leads to a significant increase in the reliability and operability of process equipment, as well as to reduce the risk of environmental and technological accidents [2].

In many cases the problem of reliable MFS operation leads to an assessment of magnetic fluid (MF) quality control, associated with its stability in the working gap of the seal. Magnetic colloids are complex objects with polydisperse particles capable of forming complex magnetostructural bonds under the influence of strong magnetic fields, which have a significant effect on the colloidal stability of MF.

The synthesis of any magnetic fluid is a compromise problem that should satisfy conflicting requirements: a relatively weak magnetophoresis of particles in a gradient magnetic field should be combined with a sufficient saturation magnetization and an acceptable value of viscosity.

Quality control of MF and its diagnostics as a complex physico-chemical system sets a number of technical problems. First of all, it is the task of selecting quality parameters for an adequate evaluation of the MF's performance in certain specific conditions.

The magnetic characteristics of MFs are the most often used ones in scientific research. However, reliable results are obtained using the expensive and narrow-profile instrumentation, which is unacceptable for arranging entrance control at enterprises operating equipment with installed MFS.

A relatively simple method for determining the sedimentation stability of MFs was proposed in [3,4], based on the measurement of the force acting on a sample with a MF in an inhomogeneous
magnetic field, as well as measuring the dynamics of the time variation of this force using high-precision electronic weights [5]. Feature of the work [4] was the conduction of experimental studies on a specially constructed gear structure sample with a MF, similar to the one that exists in MFS. Eight types of industrial MFs on different bases manufactured in LLC "Ferrohydrodynamica" have been selected as samples. However, the effect of the dimensions of magnetite nanoparticles on the performance of MFs has not been studied in this paper. High-dispersion magnetite for all eight types of magnetic fluids was synthesized by the chemical condensation method, which is based on the process of precipitation of the salts of two- and trivalent iron by a concentrated aqueous solution of ammonia (Elmore reaction [6]). The average particle size obtained by the Elmore reaction is 7...20 nm. At room temperature magnetite particles are in a superparamagnetic state and are characterized by a practically zero remanent magnetization in this range of sizes. It is the only range of particle sizes sedimentationally stable magnetic fluid can be synthesized.

The aim of this work is to study the influence of the dimensions of magnetite nanoparticles on the magnitude of the magnetic force acting on a sample filled with a magnetic fluid, and also on the character of the change of this force over time for the implementation of the express method for determining the MF sedimentation stability.

2. Research results

It is known that the physical and chemical properties of a magnetic fluid depend on the technology of MF preparation in a rather complicated manner. The MF synthesis features have an affect on this properties. They are the rate of inflow of a solution of salts of two- and trivalent iron to alkali, the intensity of mixing of the mixture, the choice of a precipitant, the removal of water, and the temperature regime. In addition, in a certain way, the properties of MF depend on the type and concentration of the stabilizer, the composition of the dispersion medium, the presence of various additives. The number of factors and features of synthesis technology that affect the properties of industrial MF is so great that it is not always possible to control them and, moreover, obtaining a fluid with given properties is often an unsolvable problem. Therefore, a magnetic fluid from one batch, but with different centrifugation times have been selected as reserch sample. Standard MF have been prepared with a centrifugation time of 1 hour, which corresponds to the technological regulations adopted in the "Ferrohydrodynamica". Also, the second part of the MF batch was centrifuged for 10 minutes, and the third batch was centrifuged for 4 minutes. Vacuum oil VM-3 (most widely used in industry), apiezon (a promising new material for a magnetic fluid), and kerosene (the properties of kerosene-based MF have been studied in detail by other authors and there is the possibility of our data comparison with the data of other researchers) have been chosen as the dispersion media.

The technical characteristics of the three samples of the magnetic fluid changed insignificantly, depending on the time of centrifugation. This is clearly seen from Table 1, where the characteristics of MFs based on kerosene are presented.

| Time of centrifugation (min) | Volumetric concentration (%) | Saturation magnetization (kA / m) | Density (kg / m3) |
|-----------------------------|------------------------------|----------------------------------|------------------|
| 60                          | 16,8                         | 60,75                            | 1315,0           |
| 10                          | 16,9                         | 57,63                            | 1312,8           |
| 4                           | 18,1                         | 59,84                            | 1358,1           |

Table 1. Technical characteristics of a kerosene based magnetic fluid with different centrifugation times.
At the same time, the yield of the magnetic fluid at the centrifugation time for 1 hour decreased by approximately 10% compared to the reduced centrifugation time, and the sediment mass was approximately 2 times greater.

The precipitate when synthesizing standard MF was thick, black tar-colored and did not spread. And when obtaining a magnetic fluid with reduced centrifugation time, the sediment consisted of two fractions (solid and liquid). The solid fraction was more friable, the liquid fraction solidified in 24 hours. The observed processes were common to all three tested liquid bases. Figure 1 shows the precipitation after centrifugation of MF based on kerosene.

Figure 1. Precipitation after centrifugation of the kerosene-based magnetic fluid at the time of centrifugation: (a) 4 min; (b) 10 min; (c) 60 min.

Changes in the formation of incompletely centrifuged MFs should be manifested in the nature of the frequency and temperature dependences of the magnetic parameters of the MF. In order to study the internal state of magnetic colloids, magnetic susceptibility is traditionally chosen as the studied magnetic characteristic. This is due to the fact that it is the most structurally sensitive parameter, in addition, the results of its study, can also provide information on the mechanism of relaxation of the magnetization of magnetic colloidal systems [7].

The magnetic susceptibility can be determined from the slope angle of the initial section of the magnetization curve. The linear initial sections for three types of MF based on kerosene with different centrifugation times, are shown in Figure 2. It follows that a decrease in the centrifugation time leads to an increase in the angle of inclination, and, accordingly, to an increase in the magnetic susceptibility. A lot of articles, detailed reviews in a number of monographs, for example, in [7-9], etc., have been devoted to the experimental determination of the frequency and temperature dependences of the magnetic susceptibility and the relaxation time for MFs based on kerosene. Their amount considerably exceeds the number of publications for MFs on more viscous bases. In order to be able to compare our results on the real and imaginary parts of the effective magnetic susceptibility with the work of other authors, they are presented for the MF based on kerosene.

The magnetic susceptibility was determined by the bridge method, described in detail in [7].
Figure 2. Linear portion of the magnetization curve for a kerosene-based magnetic fluid at centrifuge time: ● - 4 minutes, ▲ - 10 minutes, ■ - 60 minutes.

Figure 3 shows the dependence of the real part of the effective magnetic susceptibility on the temperature for two cases: in the absence of a magnetic field (Figure 3 (a)) and when a constant magnetic field is applied (Figure 3 (b)). The measurements were carried out at a frequency of 1000 Hz.

Figure 3. Dependence of the magnetic susceptibility on the temperature without a magnetic field and when applying a field for a kerosene-based magnetic fluid at the time of centrifugation: ● - 4 minutes, ▲ - 10 minutes, ■ - 60 minutes.

From their consideration it follows that the maximum values of $\chi$ when the magnetic field is applied shift to the region of higher temperatures for the MF with a reduced centrifugation time. In view of this, according to [10], MF samples that have undergone partial centrifugation have larger
particles, since the energy barrier for the particles of these samples has a higher value, and therefore their transition to the superparamagnetic state should occur at a higher temperature. The same conclusion can be drawn from an analysis of the frequency response of the imaginary part of the magnetic susceptibility presented in Figure 4, since in this case the extremum shifts toward low frequencies.

![Figure 4. Dependence of the imaginary part of the magnetic susceptibility on the frequency of the magnetic field for a kerosene-based magnetic fluid at the time of centrifugation: ● - 4 minutes, ▲ - 10 minutes, ■ - 60 minutes.](image)

So, for further research, we obtained samples of a magnetic fluid on three liquid bases (vacuum oil, kerosene and apiezon) with a centrifugation time of 60 min, 10 min, and 4 min. From the foregoing, it is clear that we can neglect the influence of random factors, depending on the production technology of MF. Therefore, we can study the character of the time variation of the magnetic force acting on the MF in the inhomogeneous field of a permanent magnet. The experiments were carried out according to the procedure detailed in [4].

To quantify the dynamic processes occurring in a magnetic fluid we use the coefficient of the magnetic force change as a diagnostic parameter, calculated as

$$K_F = \frac{\Delta F_m}{F_m(0)},$$

Where $\Delta F_m$ is the maximum change in the magnetic force at the end of the magnetic relaxation process, and $F_m(0)$ is the magnitude of the force at the initial instant of time.

The results of measuring the time-varying relative value of the magnetic force acting on samples of a magnetic fluid with different liquid bases and the centrifugation time are shown in Figure 5.
Figure 5. Dependence of the relative magnetic force on time for magnetic fluids on different bases and duration of centrifugation: _ _ _ _ - 4 min., _____ - 10 min., _ _ _ _ _ - 60 min.
From the graphs it follows that the nature of the change in the relative magnitude of the magnetic force with the passage of time is the same. For magnetic fluids that have undergone complete centrifugation, the change in the magnetic force during 10-15 minutes did not exceed 3%, which, according to the analysis in [4], indicates a high sedimentation stability of the MF.

The value of the $K_F$ coefficient is virtually independent of the reduced centrifugation time (4 and 10 min) for magnetic liquids based on vacuum oil BM-3 and apiezon, and the greatest increase in the relative magnetic force at the beginning of the experiment was different for kerosene-based MFs during centrifugation for 4 minutes. In general, MF, which has not undergone standard centrifugation for one hour, can not be recommended for long-term use as part of MFS. The remaining large particles significantly reduce the stability of the MF, and, accordingly, the reliability of the MFS.

Conclusions
1. A relatively simple method of rapid analysis of the quality of a magnetic fluid proposed in Ref. [4] was supplemented by studying the dependence of the dynamic characteristics of MFs on the size of magnetite particles.
2. Experimental samples differing in the size of magnetic nanoparticles were obtained, which was achieved by different centrifugation times of the initial MF. It is shown that the performance characteristics of these MF samples differ by not more than 10%.
3. It has been confirmed that in spite of the fact that magnetorite particles are isolated with the Elmore reaction without residual magnetization, the centrifugation stage is mandatory for obtaining a stable MF. The average particle size in the MF should not exceed 8 nm, and the change in the magnetic force acting on the sample with a MF in a given inhomogeneous magnetic field should not exceed 3% in a time of 10-15 minutes.
4. The proposed method is relatively simple and can be implemented in an enterprise operating a magnetic fluid seals to analyze the characteristics of the MF before filling it into the seal.

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