Science and technology of ferrofluids

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Ferrofluids are scientifically interesting and technologically useful materials. In this account some of the interesting properties especially those investigated in our laboratory are discussed. A few applications developed by us are also described.

COLLOIDAL dispersion of nanomagnetic particles having number density of the order of 10^{23} per m^3 is known as ferrofluid or magnetic fluid. To prevent aggregation due to van der Waal and magnetic attractions the dispersion is either sterically stabilized by coating a layer of suitable surfactant on each particle[1,2] or charge stabilized by providing negative or positive surface charge on each particle (Figure 1). When subjected to an external magnetic field, such a fluid exhibits a large magnetization and as soon as the field is removed the fluid – almost instantly – attains its zero magnetization state. Since each particle possesses a giant magnetic moment compared to paramagnetic particles such a medium is also called superparamagnetic, i.e. having zero remanence and no coercivity. A gradient magnetic field generates an additional body force, the ferrohydrodynamic force ($\mathbf{M} \cdot \nabla \mathbf{H}$), which modifies hydrodynamics of such fluids[3]. Several new phenomena arise because of this body force: the fluid can be suspended in space by applied magnetic field, stable levitation of nonmagnetic as well as magnetized materials, generation of fluid motion by thermal or magnetic means without any external moving parts, ability to flow and conduct magnetic flux, and spontaneous formation of stable liquid spikes in presence of a perpendicular magnetic field (Figure 2). Thus the combination of these fluidic and magnetic properties is exploited in many innovative engineering devices like rotary shaft seals, dampers and centrifugal switches[4,5]. More than 2500 patents exist in this field. It is speculated that the biggest application of this fluid is yet to come and what we see presently is just 'the tip of an iceberg'. A ferrofluid is not only technologically important, but it is scientifically also equally interesting. Thus as stated above, several novel phenomena related to hydrodynamics of ferrofluids have been discovered, and consequently it is possible to simulate and study certain condensed matter phenomena like gas–liquid transition, fractals, dipolar glasses and pattern formation. In the following sections preparation methods of ferrofluids, certain physical and magnetic properties of these fluids, and a few applications of these fluids are discussed.

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Preparation of ferrofluids

In nature, some of the aquatic bacteria orient and migrate along geomagnetic lines, a phenomenon called magnetotaxis[6]. This is because the bacterial cell exhibiting the magnetotactic property shows presence of single-domain magnetic particles coated with layers of fatty acid(s). Barring this example, ferrofluids are not found in nature. To synthesize ferrofluids at least two components, monodomain magnetic particles and a suitable carrier liquid, are required. Since randomizing Brownian energy may not be enough to counteract attractions owing to van der Waal and dipole–dipole forces, aggregation and sedimentation are prevented by providing suitable repulsive forces either by Coulomb repulsion or by steric repulsion. While in the former case particles are either positively charged or negatively charged and the fluid is called ionic ferrofluid, in the latter case each particle is coated with an appropriate surfactant and the resulting fluid is classified as surfacted ferrofluid. Whereas the ionic ferrofluids require a polar medium like water as the carrier, the surfacted ferrofluids can use any carrier liquid like oil, water and hydrocarbon. By far a great deal of work has been carried out for surfacted ferrofluids. Several methods are available for the synthesis of monodomain magnetic particles[3]. Each method has its own characteristics. Size reduction of coarse magnetic particles by ball milling was first used by Papell and modified

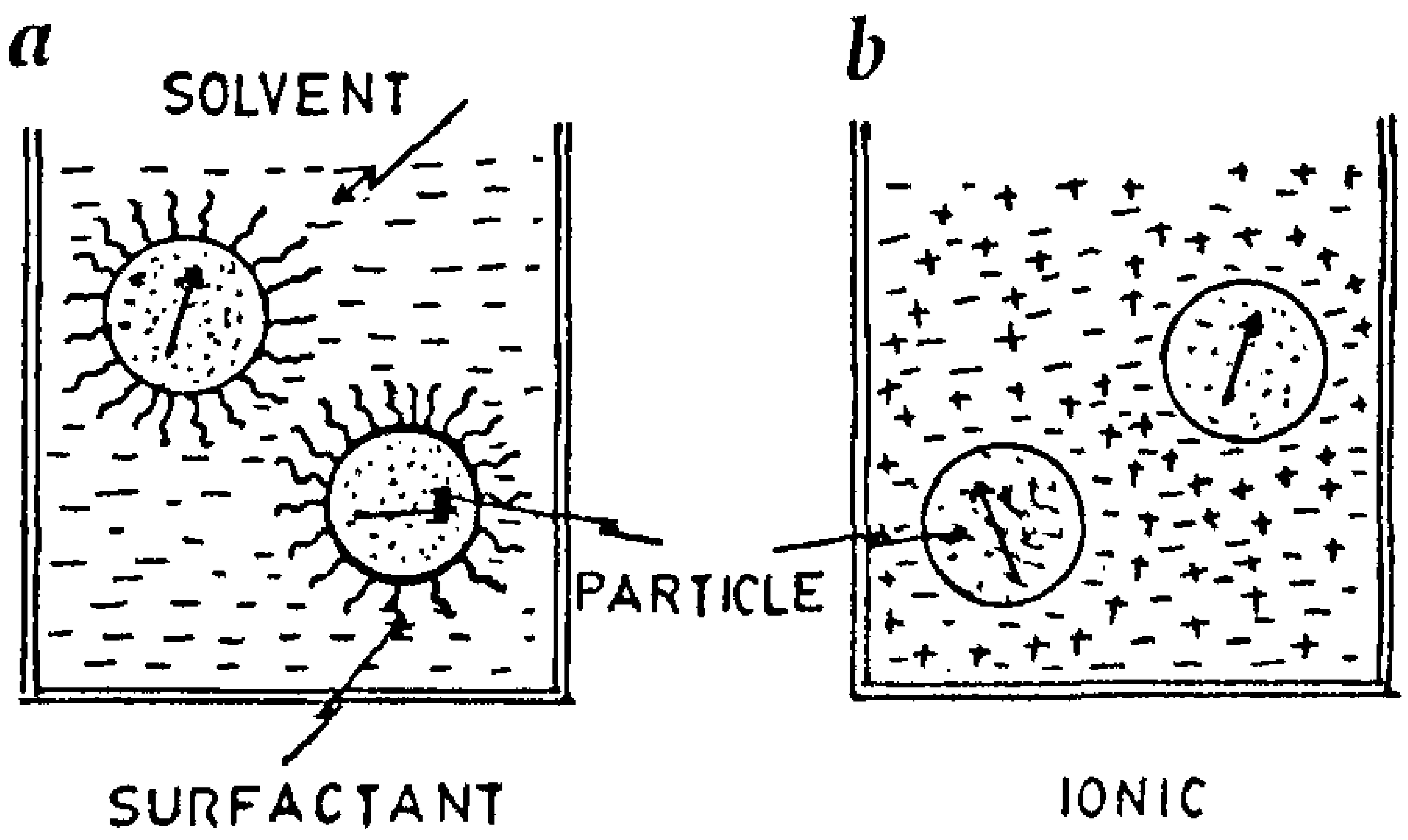

Figure 1. Surfacted and ionic ferrofluid.

by Rosensweig *et al.*[7]. Though this method is still popular, it requires a prolonged (months) grinding time and, further, particle size distribution is broad. Co-precipitation of ultramicroscopic magnetic particles from salt solutions is suitable for rapid production but control of pH and temperature, and purity of chemicals are essential to maintain their stoichiometry. The possibility of occurrence of free radicals and large surface anisotropy of the particles further complicate the physics of ferrofluids. However new recipe of monodomain magnetic particles may easily be formulated by this method.

Fe–Mn ferrite particles have been prepared by the co-precipitation technique. A mixed solution of 0.1 M in $MnCl_2 4H_2O$, 0.9 M in $FeSO_4 7H_2O$, and 2 M in $Fe(NO_3)_3 9H_2O$ was prepared. A 25% NH_4OH solution was added to the above solution until the pH reached 9.5. Oleic acid (5 ml) was added to the solution which was heated for 5 min at 95°C. The pH was maintained at 9.5 during the heating process. To coagulate the oleic acid-coated particles, an acid solution was added. After decantation of the solution, the resulting product was washed many times with distilled water to remove impurities and free radicals. Finally water was removed by washing with acetone. This acetone-wet slurry was dispersed in 20 ml of household-grade kerosene and stirred for 20 min at 60°C. The resulting fluid was centrifuged at 12000 rpm for 30 min. Figure 3 shows the flow chart of this method[8]. Over and above the conventional ferrofluids involving magnetite particles, several new ferrofluids involving partially substituted Mn–ferrite particles, Mn–Zn ferrite particles, Gd-doped particles have been synthesized in this laboratory[8–10]. All these ferrofluids exhibit several interesting features. With Gd-doped fluids a large pyromagnetic coefficient[10] could be obtained, while with the partial substitution of Mn ferrite a moderately high gauss fluid with low viscosity could be produced[8]. Using the Mn–Zn ferrite particles, it was possible to synthesize a low Curie temperature ($T_C = 340$ K) fluid and by recording of small angle neutron scattering (SANS) patterns below and above Curie temperature, for the first time it has been possible to segregate contribution due to nuclear and magnetic scattering of using unpolarized neutrons[9]. Some of these results are described in the next section.

Choice of the carrier liquid depends on the application. For example: (i) For rotary shaft seal in vacuum systems low vapour pressure, carrier-like silicone or diester oil should be used. (ii) For inclination sensor hydrocarbon, carrier-like kerosene is adequate[11–13]. (iii) For sink–float separator, water may be used[3].

For surfacted fluids, selection of surfactant is crucial for its stability. A surfactant molecule consists of a polar head and a tail of hydrocarbon chain, e.g. oleic acid

$$CH_3(CH_2)_7CH\text{–}CH(CH_2)_7C\begin{smallmatrix}\nearrow OH \\ \searrow O\end{smallmatrix}$$

The anchor polar group is adsorbed on the particle while the chain performs thermal movement in the carrier. When a second particle with similar chain approaches closely (Figure 1), the movement of the chains is restricted and result in steric repulsion. A simplified estimate for the entropic effect for short chains is given by Rosensweig[3]

$$E_s = \frac{2}{3}\pi N k_B T \left(\delta - \frac{x}{2}\right)^2 \frac{\left(1.5D + 2\delta + \frac{x}{2}\right)}{\delta}, \qquad (1)$$

where N is the number of molecules anchored per unit area, δ is the thickness of the stabilized layer, x is the

Figure 2. Ferrofluid spikes observed in a perpendicular magnetic field.

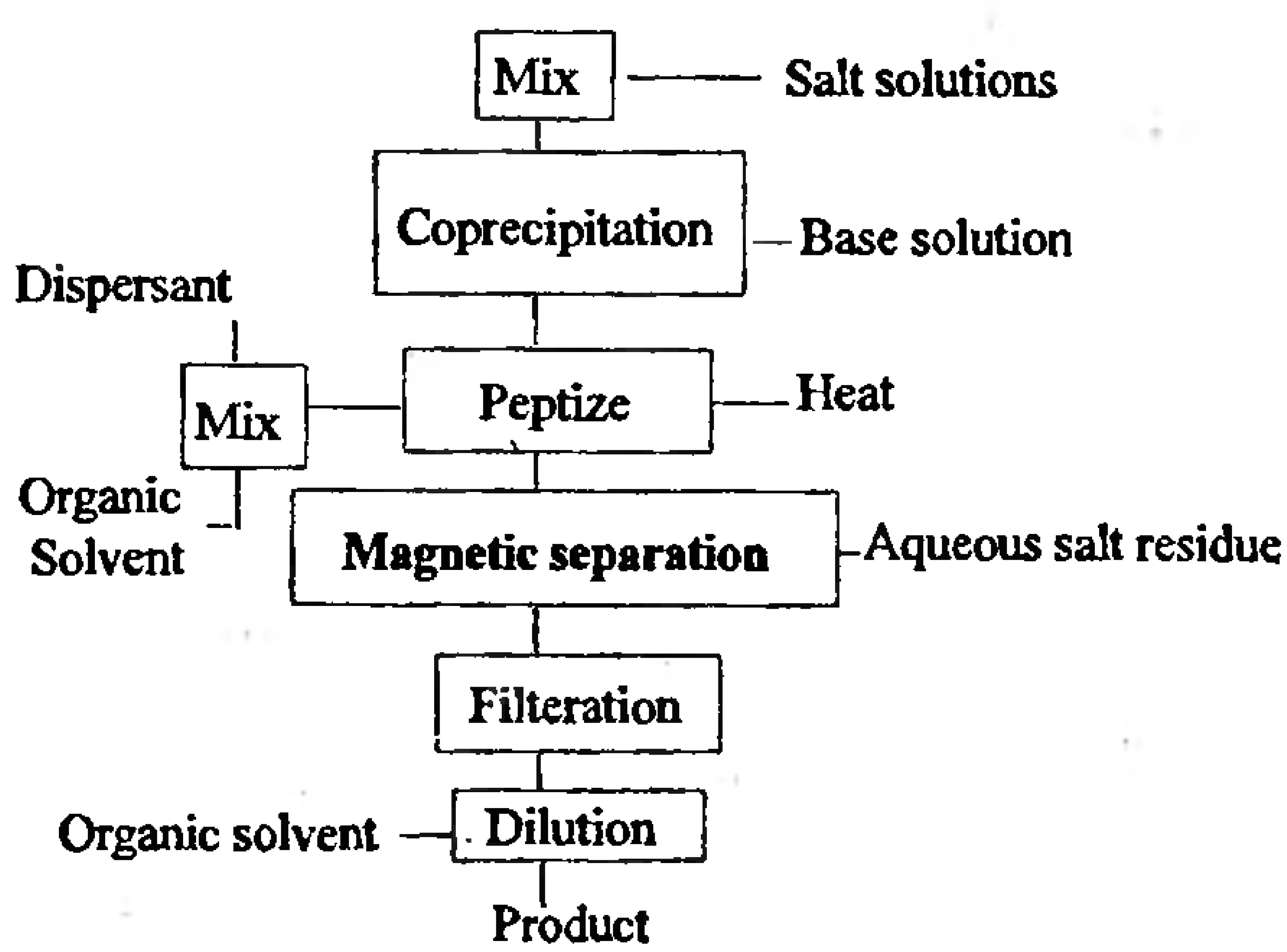

Figure 3. Flow chart of preparation of ferrofluid by the co-precipitation technique.

distance between two surfaces, and D is the diameter of the particle. This equation is useful to calculate δ which gives a reasonable barrier of $25k_BT$ to prevent agglomeration.

In case of ionic magnetic fluids, the surface electric charge promotes electrostatic repulsion between the particles via the solvent–particle interaction. Balance of attractive and repulsive forces can be governed by this charge. Stable dispersion in case of γ-Fe_2O_3 particles is obtained if pH is around 2 and ionic strength $\approx 5 * 10^{-2}$ M (ref. 14). On raising pH the surface charge density (σ_s) decreases, the repulsion reduces, and the degree of agglomeration increases. This aggregation is limited if pH < pzc (point of zero charge). At pH $\approx$ pzc, $\sigma_s = 0$ and a floc forms. At pH $\geq$ pzc, once again the particles redisperse but are susceptible to aggregation[15].

Thus both for surfacted as well as ionic ferrofluids care should be taken to synthesize magnetic particles, either by coating these particles with a suitable surfactant or assign an appropriate (positive or negative) charge and disperse them in the carrier liquid. It may be noted that though metallic particles like iron, cobalt, or nickel may be used for ferrofluids, oxidization, toxicity, etc. limit their usefulness.

Characterization

Structure

Fe^{3+}-based spinel ferrite particles are the most popular in the synthesis of ferrofluids. When co-precipitation technique is used to prepare such particles, their spinel structure has to be ascertained. X-ray diffraction study of the dried powder of the fluid is the most convenient way to decide about their crystal structure. X-ray diffraction pattern of Mn–Zn sample is shown in Figure 4 (ref. 9). Using Rietvield refinement programme, particle size, lattice parameter, oxygen parameter, goodness of fit, and cation distribution can be precisely determined[9]. If presence of ferrihydrite phase or, because of Mn^{3+}, occurrence of tetragonally-deformed phase coexist then this can be detected by XRD. The single-phase spinel structure can also be confirmed by neutron diffraction. Cation distribution can be determined from the X-ray intensities of the peaks. Stoichiometry is confirmed by atomic mass absorption spectra, for example in case of $Mn_{0.5}Zn_{0.5}Fe_2O_4$ particles synthesized for ionic fluids it was found that Fe/Mn and Fe/Zn ratio is greater than 4. This indicated a loss of Mn and Zn during synthesis[16]. Such a loss may create a lattice vacancy, a deformation of structure, or both, leading thereby to modification of magnetic properties like the domain magnetization, M_d, Curie temperature, T_C, etc.

Size and size distribution

Particle size and size distribution in a ferrofluid may be determined by several methods: small angle neutron scattering (SANS), small angle X-ray scattering (SAXS), transmission electron microscopy (TEM), magnetization measurement, and viscosity. Each technique has its own characteristic, for example while viscosity measurement is sensitive to peak size diameter[17], birefringence relaxation is sensitive to tail of the distribution[18]. For certain techniques (e.g. scattering, magnetization), it is convenient to assume a priori distribution, and log-normal distribution is found to be more suitable for many ferrofluids[17]. Two parameters—median diameter D_0 and the standard deviation σ—are used to generate the log-normal distribution curve. These two parameters are determined from experimental measurements. A technique that is based on magnetization measurement is described

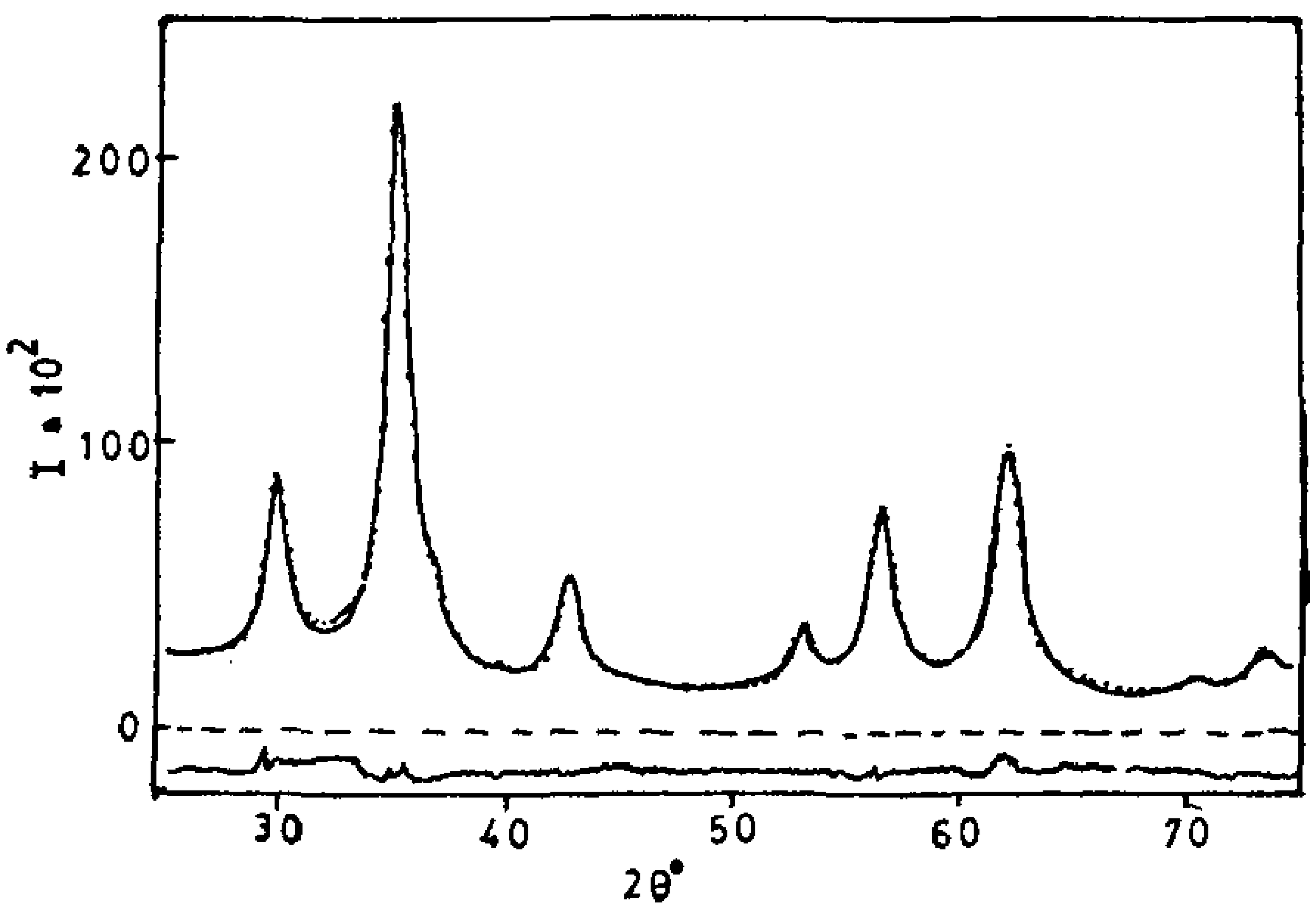

Figure 4. X-ray diffraction pattern of Mn–Zn ferrofluid.

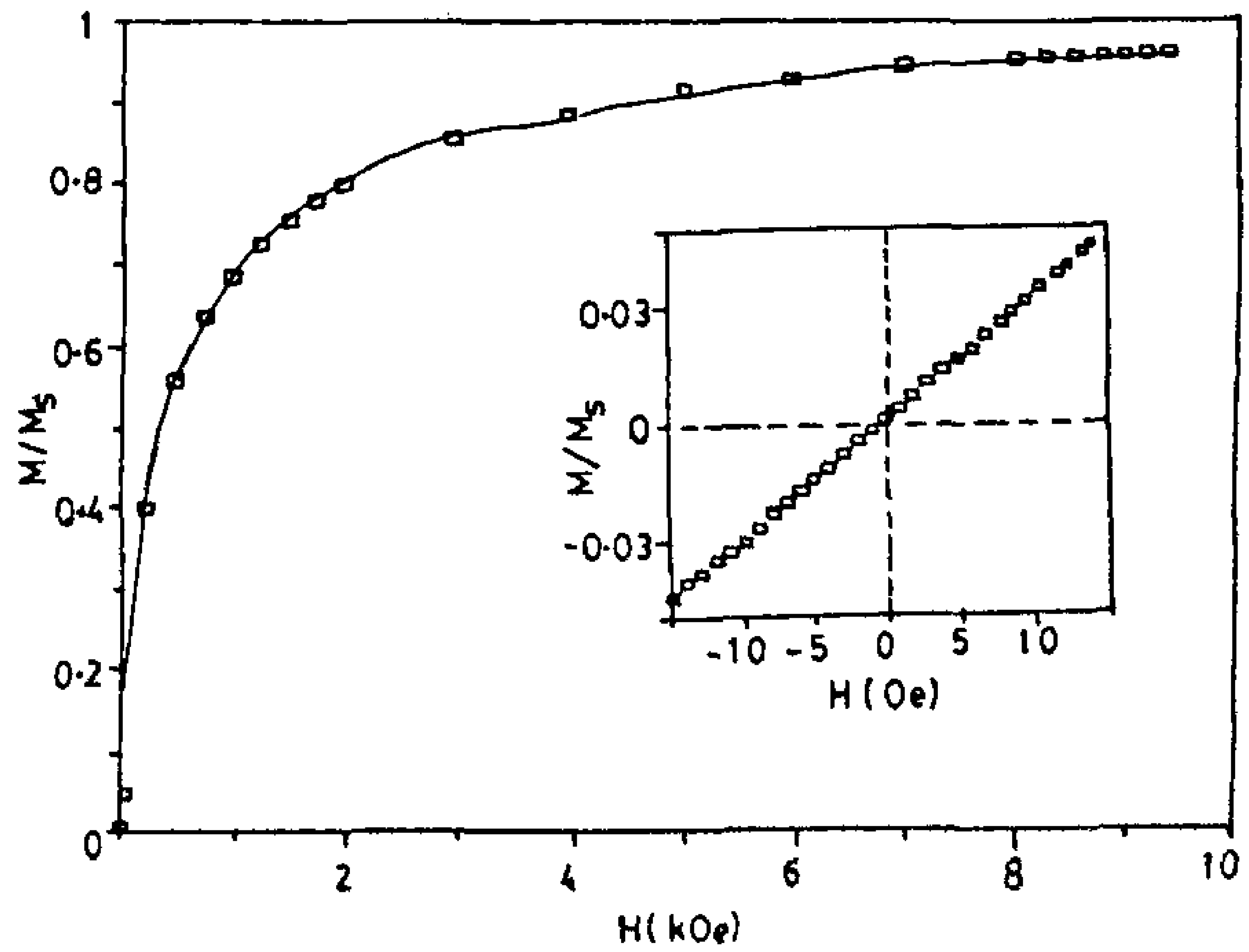

Figure 5. Variation of magnetization of the Mn-substituted ferrofluid with applied magnetic field. *Inset*, Low field magnetization curve.

here, while other techniques are described elsewhere[17,19,20].

Figure 5 shows the variation of magnetization of the Mn-substituted fluid. While the larger particles of the polydispersed dispersion are oriented at comparatively lower magnetic field, the finer particles are oriented at higher fields. It can be shown that the particle size determined from the experimental data at the weak field strength will give the average size of the particle as $\overline{D^2}/\overline{D}$, and that at high field the average size of the particle will be $\overline{D}$. The two moments of the distribution can be calculated from the above averages as:

$$\frac{1}{2\sigma^4}-\frac{1}{\sigma^2}=\frac{1}{2}\left[\frac{\overline{(D)^3}}{((\overline{D})^2+\overline{D})^3}+\overline{(D)^3}\right], \tag{2}$$

and

$$\overline{D}=D_0\,\exp\left[\frac{\sigma^2}{6}\right]. \tag{3}$$

The log-normal distribution is given by

$$f(D)\,\mathrm{d}(D)=\frac{1}{\sqrt{2\pi}\,\sigma D}\,\exp\left[-\frac{(\ln D-\ln D_0)^2}{2\sigma^2}\right]\mathrm{d}D. \tag{4}$$

Here it is assumed that domain magnetization, M_d is known and that it remains constant for the range of distribution. In the present case, $\overline{D}$ and $\overline{D^2}/\overline{D}$ were found to be 129 Å and 139 Å, respectively, yielding $\sigma=0.67$ and $D_0=120$ Å. The appropriate log-normal size distribution curve obtained for Mn-substituted ferrofluid is shown in Figure 6. It was found that though D_0 determined by SANS, SAXS or magnetization measurements is almost the same, σ were differed. This may be due to truncation of the data after certain angle or field.

Magnetization

Study of variation of magnetization of a ferrofluid as functions of applied field (dc or ac) and temperature is useful not only for its application but also to understand the behaviour of nanomagnetic particles.

In a ferrofluid, each particle with its embedded magnetic moment m resembles a paramagnetic molecule. Under equilibrium condition the tendency of the dipoles to align along the applied field is partially overcome by thermal agitation. If interaction between the particles is neglected, Langevin theory gives magnetization M of the fluid on the basis of

$$M=\phi\, M_d\, L(\alpha)\,, \tag{5}$$

where ϕ is the volume fraction of particles, and $L(\alpha)$, with $\alpha=mH/k_BT$, is the Langevin function $(\coth\alpha-1/\alpha)$. Log–log matching of theoretical plot of $L(\alpha)$ vs α and the experimental plot of M vs H gives a fairly accurate value of saturation magnetization M_s of the fluid. As mentioned earlier, the two moments of the size distribution can also be determined from this matching[17,20].

It was found that variation of initial (ac or dc) susceptibility (χ_i) with temperature cannot be accounted by Langevin theory and that interparticle interaction has

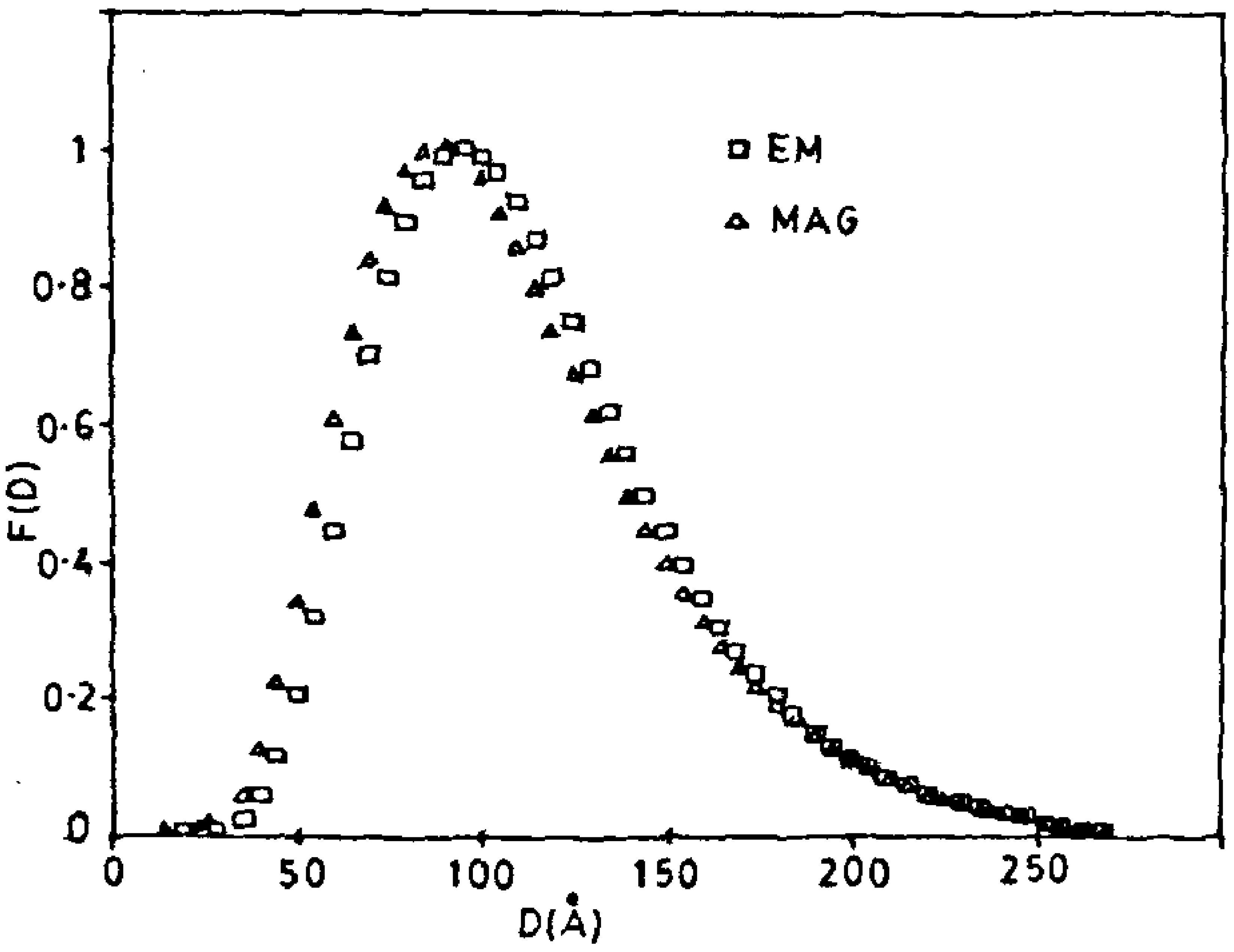

Figure 6. Log-normal size distribution curve obtained for Mn-substituted ferrofluid. EM refers to TEM measurements and MAG refers to magnetization measurement.

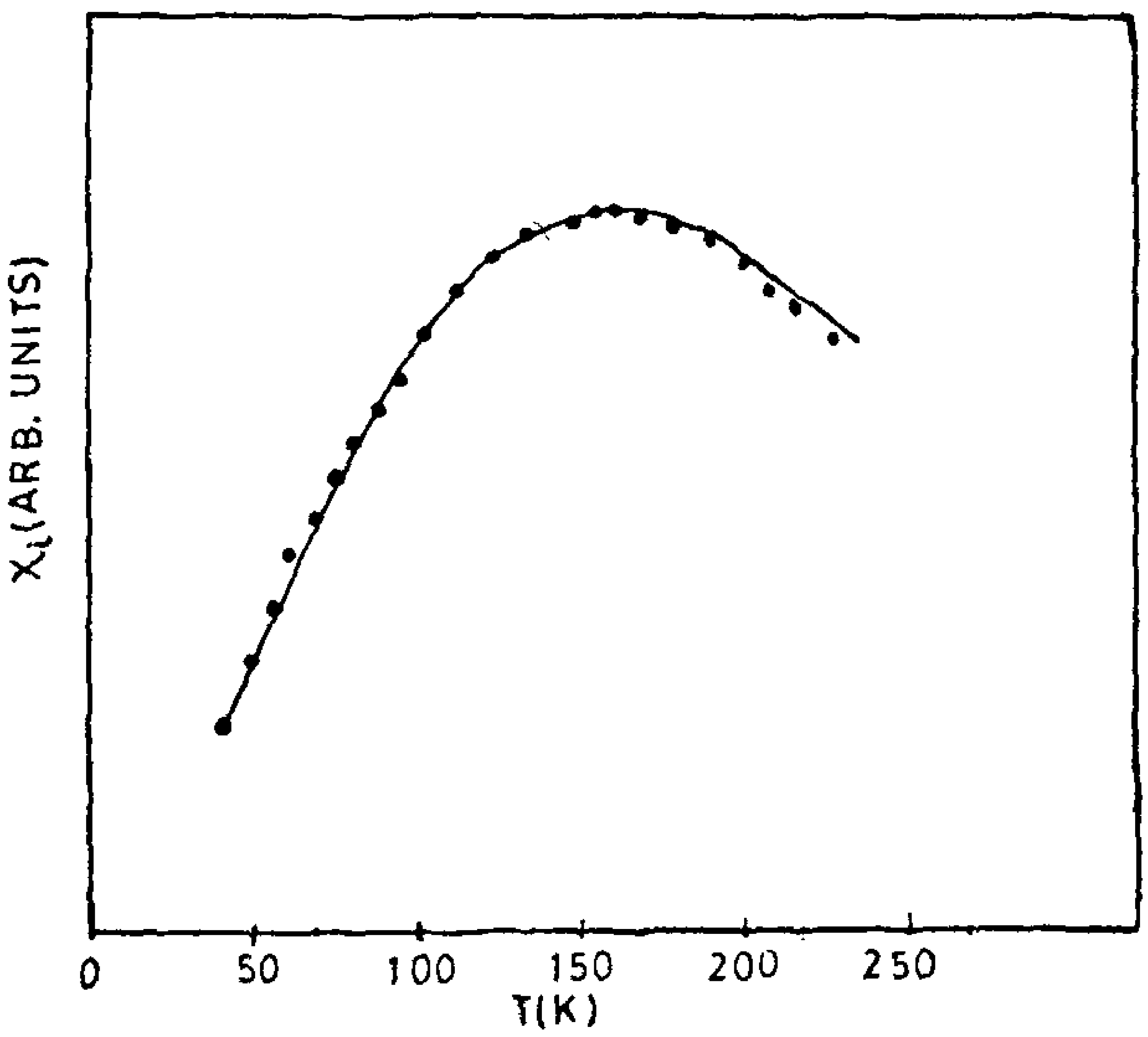

Figure 7. Variation of initial ac-susceptibility as a function of temperature for Mn-substituted ferrofluid.

to be incorporated. Using mean spherical approximation, Shliomis was able to explain this dependence[21]. a–c susceptibility will detect only the reversible part of the total magnetization. For fine particles the irreversible magnetization corresponds to the magnetization of the 'blocked particles', i.e. the particles that are unable to overcome the energy barrier arising out of magnetocrystalline and/or shape anisotropies. The particles which cannot relax over this barrier to attain equilibrium are responsible for irreversible part of the magnetization. If all the particles are of the same size, ac-susceptibility curve will show a sharp peak at the blocking temperature. The observed broad peak (Figure 7) is due to a spectrum of blocking temperatures arising from the size distribution of particles. Once again log-normal distribution of particles can be assumed to explain this observation. The upper limit on the particle volume for superparamagnetism[22] (SP) will be[22]

$$V_{sp} = 17k_BT(M_s/K)\,. \qquad (6)$$

Since the contribution due to the blocked particles will be negligibly small, only those particles for which $V < V_{sp}$ will contribute to the ac-susceptibility. Therefore magnetization due to these SP particles is given by

$$M(T) = N\int_0^{m_p} L(\alpha)\,F(m,\sigma)\,\mathrm{d}m\,. \qquad (7)$$

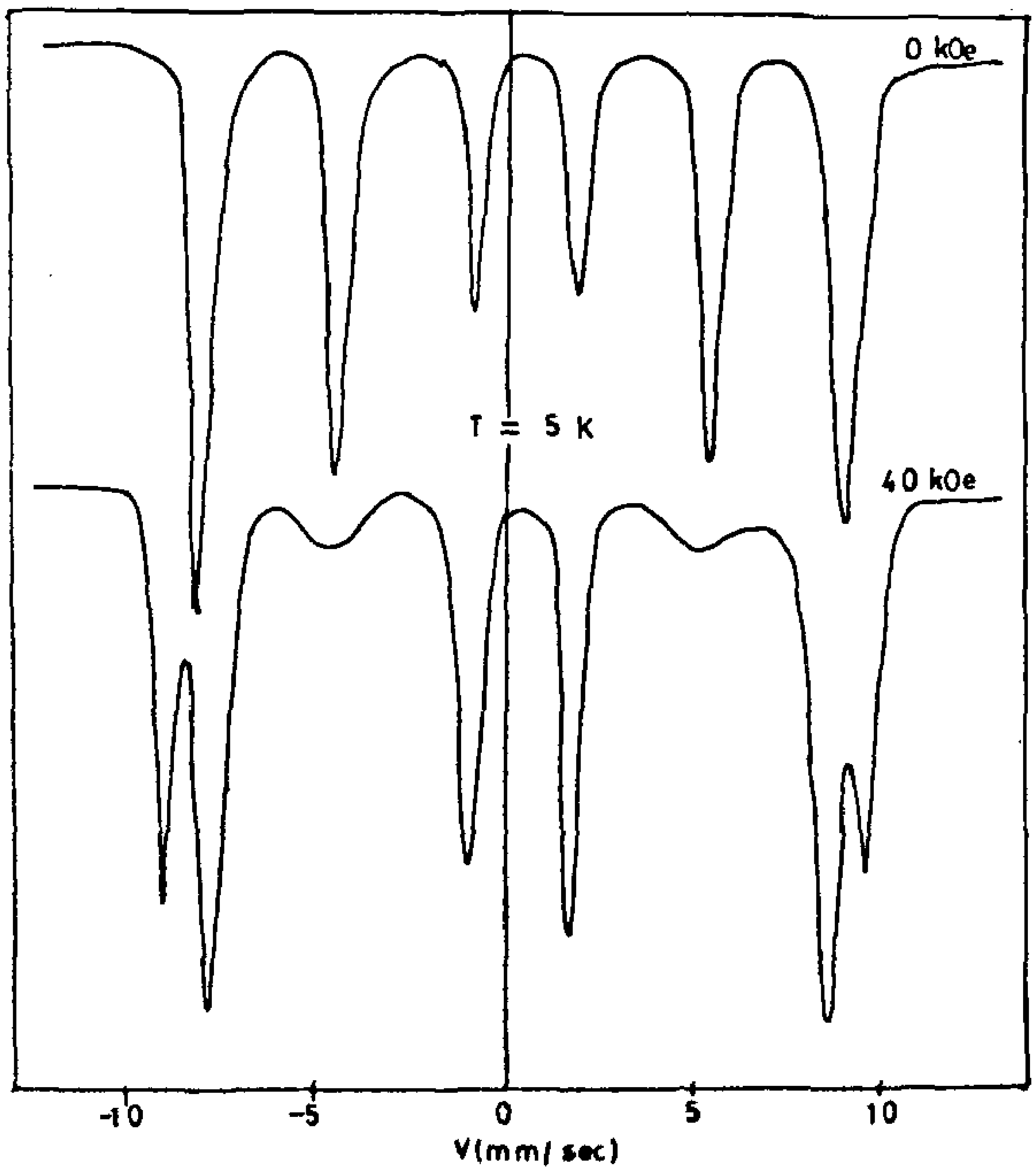

Figure 8. Mossbäuer spectra at 5 K for Mn-substituted ferrofluid.

In the low field limit, equation (7) gives the following:

$$\chi_i(T) = \frac{M_s M_d V_0}{3k_BT}\exp[1.5\sigma^2]\int_0^{V_{sp}}\frac{1}{\sqrt{2\pi}}\exp\left[-\frac{U_p^2}{2}\right]\mathrm{d}U\,, \qquad (8)$$

and

$$U_p = \frac{1}{\sigma}\left[\ln\frac{17k_BT}{KV_0} - 2\sigma\right]. \qquad (9)$$

$d\chi_i(T)/dT$ gives a peak at T_p for which $U_p = 0$. Solid line in the Figure 7 shows the fit to the experimental points. The parameters for this best fit are $D_0 = 120$ Å and $\sigma = 0.5$, $K = 2.75\times10^5$ erg/cc. These results are in agreement with those reported earlier[20].

Temperature dependence of dc-susceptibility of a ferrofluid can easily be measured with Quincke's method. This method is particularly well suited to determine Curie temperature of low-Curie-point-temperature sensitive ferrofluids[23]. Using this method, Curie temperature (T_C) of Mn–Zn, Mn–Fe, and Gd–Mn–Zn ferrofluids was determined[23,10] and the obtained values agreed with those determined by other methods.

Nanomagnets in a ferrofluid are free to rotate under Brownian mechanism. Hence even if the magnetic moment is rigidly coupled with the particles, its moment will rotate due to this mechanism. Relaxation time for this is given by $\tau_B = (3V\eta_0)/(k_BT)$, where η_0 is the carrier viscosity. This extrinsic SP is quite different from the intrinsic SP earlier postulated by Neel, wherein magnetic moment of solid particle reverses with the reversal of the field. Unlike Brownian mechanism, no mechanical rotation of the particles is involved here. Neel relaxation time is given by $\tau_N = (1/f_0)\exp(KV/k_BT)$, $f_0 = 10^9$ Hz. If $KV/k_BT < 20$ then the particle is termed as intrinsic SP. Typical values of τ_B and τ_N are 10^{-7} sec and 10^{-9} sec, respectively. Hence at normal temperature and low frequency, the extrinsic SP state is dominant. In high

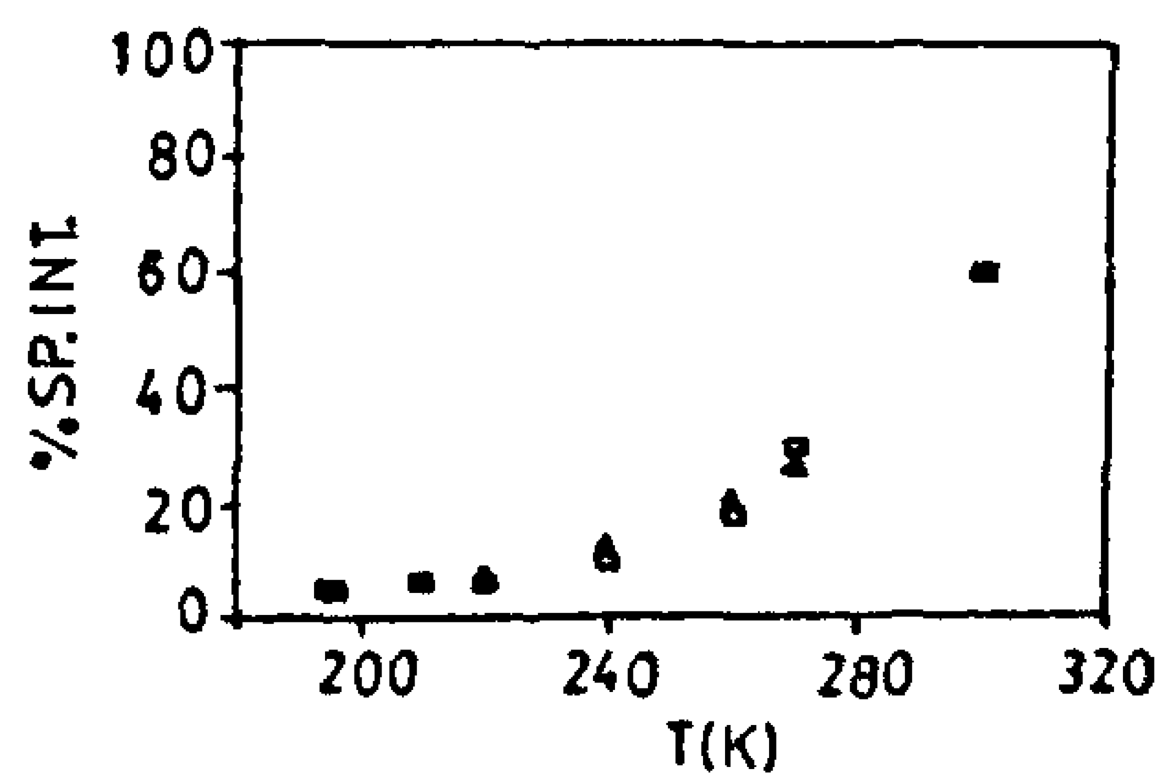

Figure 9. Percentage change in intensity of SP phase with temperature.

frequency region, the moment detaches from the anisotropy axis and moves inside the crystal. Mossbäuer and ESR spectroscopy are utilized to distinguish between the two types of superparamagnetism.

Mossbäuer spectroscopy is extensively used for studies on magnetic properties of microcrystals. By lowering the temperature, magnetic phase transition from SP to Ferromagnetic phase can be studied. Temperature at which this transition occurs is also called the blocking temperature, T_B. In ferrofluid, usually microcrystals are coated with surfactant which is chemiabsorbed on the surface of the particle. When size of the crystals are below 100 Å, surface anisotropy is dominant. Role of the chemisorption of surfactant on surface anisotropy is studied by Mossbäuer spectra of coated and uncoated particles[19,24]. It was observed that the domain magnetization of the coated particle was lower than that of bulk value. To account for the loss of magnetization, it was postulated that a dead layer of 8 Å may have been formed on the core of particles. However in such a case, the central absorption line should have covered more than 50% of the spectra. But the spectra were almost identical with or without coating[24]. Figure 8 shows the Mossbäuer spectra at 5 K for Mn-substituted fluid[19]. In zero field, the six lines show a slight asymmetry. When a field of 40 kG was applied parallel to the direction of γ-rays, areas of lines 2 and 5 substantially decrease. The intensity of lines 2 and 5 suggests that a small fraction of the atoms are not magnetized in the field direction. This fraction was estimated to be 8.5%. This may be due to noncollinear spin structure near the surface which usually occurs in fine particles. The splitting of the lines 1 and 6 into two components in the field of 40 kG indicates that the field is parallel or anti-parallel to the two sublattices. The canting angle was calculated to be 75° (ref. 25) and considering the canted spin fraction of 8.5%, M_s value was found to be 90 emu/g, which is quite close to 92 emu/g of bulk. Hence it was concluded that the observed low value of magnetization is due to the surface pinning effect.

Electron spin resonance technique has emerged as a complimentary tool to study the kinematics of magnetization in a ferrofluid[26–28]. From the spectra recorded at different temperatures and dilutions the linewidth (ΔH), g-factor and resonance field H_r can be deduced. These data are used to draw inferences like chain formation, phase transition and anisotropy factor[28,29].

A few examples of the results of Mn–Zn surfacted ferrofluid and Gd-doped Mn–Zn surfacted fluid are discussed in the following section.

ESR spectra of certain ferrofluids exhibited the three-line structure, i.e. a broad line coming from ferromagnetic particles along with a broad shoulder at $g = 4$ and a sharp line at $g = 2$ (refs 26–28). Such a feature may arise due to the following reasons: (i) The observed line at $g = 4$ may be due to magnetic anisotropy or due to the Fe(III) complexes attached to the surfactant molecule. (ii) The line at $g = 2$ may either be due to free Fe(III) radical or due to presence of intrinsic superparamagnetic particles. Absence of such a sharp line at low temperature may be due to the increased linewidth of ferromagnetic particles masking the sharp line. However since no change in intensity was observed, it was difficult to conclude about the cause of this line. The main reasons for this are the following: (i) Magnetic anisotropy of particles usually used in ferrofluid synthesis is large and saturation magnetization of the fluid does not vary much with the temperature in the range of 200 to 400 K. Further, Curie temperature of such fluid is high, hence for a polydispersed system fractional change of SP phase will be quite small. This difficulty can be overcome by the temperature-sensitive Mn–Zn fluid having Curie temperature 340 K. ESR spectra of this fluid showed that T increases the contribution due to SP phase increases for the line $g = 2$. It was 5% at 200 K and 60% at 300 K (Figure 9) (ref. 30). SANS results also indicated similar variations[9]. Thus $g = 2$ line is due to SP phase. Similarly from the analysis of ESR spectra of frozen chains it was concluded that the line at $g = 4$ in the sample is due to Fe(III) complex attached to surfactant molecule[28].

Gd-doped Mn–Zn ferrofluid is also a temperature-sensitive fluid having large pyromagnetic coefficient and Curie temperature 348 K. ESR spectra of this fluid exhibited anisotropy for field-cooled sample and on increasing temperature intensity resonance field and linewidth, exhibited a large change[10]. The analysis showed that there is a transition at 341 K close to the Curie temperature of this fluid. Thus ESR study helps to probe the mechanism and kinematics of magnetization in a ferrofluid. It may be mentioned here that these informations are not only academically interesting but also help to design devices based on ferrofluids like gyroscope, pumps etc.

Other physical properties like FTIR, ultrasonic and optical properties also provide useful information on ferrofluids[18,21,31]

Applications

As mentioned earlier, a large number of applications have been developed involving one or more novel characteristics of this fluid. List of patents has been published in the special issue of *Journal of Magnetism and Magnetic Materials (JMMM)* covering proceedings of International Conferences on Magnetic Fluids[32]. We shall describe here only a few of these applications developed in this laboratory.

Seals

Rotary shaft seal is the most commercially exploited application of ferrofluids[4]. Between stationary housing and rotating shaft, 'O' ring(s) of ferrofluid is formed by a ring magnet and focusing pole structure. An 'O' ring can sustain a certain pressure difference which depends on saturation magnetization of the fluid, field gradient generated by the permanent ring magnet and pole structure[12]. Such seals are used in rotating anode X-ray generator, disc-drive system for computer, etc. A simple computer program has been developed, which may be used for simulation study of the magnet dimension, focusing structure, the aspect ratio, i.e. the ratio of the flux density in active and inactive gap, the number of stages and the pressure capability of the seal[33].

Inclination sensor

As stated earlier, a ferrofluid can transmit and conduct magnetic flux and may be used as a soft magnetic core. This property is used in designing a ferrofluid inclinometer. Compared to the conventional devices, such a sensor is less prone to be affected by environment and is more sturdy, therefore may be used in avionics, robotics, machine tools, etc. The sensor based on variable inductive core has been designed and fabricated[13,34] Using simple construction and at low cost, it was possible to achieve an accuracy of 6 sec of arc. Variation of sensitivity as a function of frequency, inclination, input voltage and fluid level could be explained on the basis of simple theory.

Biotechnological applications

It has been demonstrated that nanomagnetic particles of ferrofluids are useful in the field of biotechnology[35,36]. Magnetic particles coated with a suitable stabilizer like dextran, saline or heparin were used to bind cells from whole blood for radioimmune assay (RIA). A new method of binding protein molecule directly on freshly prepared magnetite has been developed[37,38]. The binding was confirmed by TEM, magnetization measurements, and FTIR. Under optimum conditions, more than 90% of the protein was bounded to the magnetic particles. Other antibodies, enzymes and cells were also used for this purpose[39].

Centrifugal switch

Centrifugal switches are used to cut off primary windings after the rotor of the motor attains certain speed. The conventional switch has a large inertia and special care is required for its use in explosive atmosphere. A ferrofluid-based switch involving levers and reed switch was developed earlier[40] but it has a dead zone area of nearly 600 rpm. A switch based on differential transformer with ferrofluid core has been developed in this laboratory which has almost zero dead zone, explosive-proof and very low inertia[41].

Dampers

A permanent magnet immersed in a pool of ferrofluid levitates itself from the bottom. This property is used in inertia dampers[4]. Similar damper for stepper motor has also been developed[42].

This paper briefly described some of the aspects of science and technology of ferrofluids. Several other aspects like ferrohydrodynamical asymmetrical stress, instabilities, composites are equally interesting. Despite more than 3000 papers published in this field, this field still offers opportunities for pure as well as applied research.

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