Relevance of magnetic moment distribution and scaling law methods to study the magnetic behavior of antiferromagnetic nanoparticles: Application to ferritin

N. J. O. Silva,* V. S. Amaral, and L. D. Carlos
Departamento de Física and CICECO, Universidade de Aveiro, 3810-193 Aveiro, Portugal

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The analysis of magnetization of antiferromagnetic nanoparticles is not straightforward due to the presence of a linear component, $\chi_{AF}H$ superimposed on the saturation and the inexistence of a simple relation between size and magnetic moment μ. We present a method, based on scaling laws, to determine the variation of χ_{AF} with temperature and to find the temperature dependence of $\langle\mu\rangle$, without any assumption on both the magnetization dependence on the magnetic field and the moment distribution function. We have applied this method to ferritin nanoparticles (with very narrow size distribution) and found that, independently of the magnetization law, $\langle\mu\rangle$ decreases with increasing temperature and that a magnetic moment distribution function cannot be ignored. The fit of the magnetization data with Langevin and lognormal moment distribution functions yielded $\langle\mu\rangle = 120\ \mu_B$ (at 30 K), decreasing to about 70% of this value at $T = 250$ K, in agreement with the scaling method estimations, and a log(μ) variance $s^2 = 1$. This result shows that in ferritin there is no direct relation between size and moment distribution and that disorder should play a major role in the moment distribution. In general, if a moment distribution is ignored, the fitted magnetic moment presents an artificial systematic increase with temperature, similar to some previous reports in the literature. This highlights the need for evaluating the effect of such a distribution before drawing conclusions about the physical nature of the parameters variation.

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I. INTRODUCTION

Magnetic nanoparticles are of much interest due to their application in high density magnetic storage media and emergent applications in biomedicine as magnetic cell sorting and magnetic fluid hyperthermia. Finite-size and surface effects dominate the magnetic properties as size decreases, leading to unusual properties, distinct from bulk material. While in the framework of superparamagnetism[1] the magnetic properties of ferromagnetic nanoparticles are well understood, antiferromagnetic nanoparticles show further peculiarities, namely the dependence of magnetization with size and temperature, not yet explained.

The magnetization of antiferromagnetic nanoparticles arises from uncompensated/canted spins, and their number depends on size and on disorder. Unlike the case of ferromagnetic particles, in antiferromagnetic particles the dependence of magnetic moment with volume is not straightforward, as discussed by Néel.[2] Based on Langevin analysis, several studies above a blocking temperature T_B[3–6] found that the magnetic moment of antiferromagnetic systems μ_p increases with temperature, unlike the expected decrease. This increase was then used to model microscopic characteristics,[7] derive deviations from the Curie law,[8] and was recently attributed to thermoinduced magnetization.[9] In these studies, $\mu_p(T)$ was obtained above T_B by fitting the total magnetization of the antiferromagnetic nanoparticles to the sum of a saturation Langevin component (associated with uncompensated moments) and a bulklike linear term[3,7]

$$M(H,T) = m_0 L(\mu_p H/kT) + \chi_{AF}H, \tag{1}$$

where $L(x) = \coth(x) - 1/x$ is the Langevin function, m_0 is the saturation magnetization, and χ_{AF} is the antiferromagnetic susceptibility. The saturation magnetization is equal to the product of μ_p with the number of particles per volume or mass (depending on the M dimensions), N. In nanoparticles systems, volume distributions are ubiquitous and several works consider size or moments distribution in the magnetization curves analysis.[10–13] Further deviations to this simple Langevin behavior occur due to mechanisms such as interparticle interactions[14,15] and anisotropy.[16] In antiferromagnetic nanoparticles, the relevance of these deviations is not yet fully understood.

In this work we analyze the magnetization curves of ferritin, a biological system where the iron oxide hydroxide ferrihydrite ($FeOOH.nH_2O$) nanoparticle core with ~5 nm of diameter and narrow size distribution is wrapped up in a protein template.[17] In Sec. II A, we compare parameters obtained after fitting the magnetization curves of ferritin with and without a moment distribution and we show that the parameters variation with temperature is drastically different. In Sec. II B, we present a method to separate the bulklike antiferromagnetic and the superparamagnetic components in antiferromagnetic nanoparticles that allows us to estimate the dependence of the magnetic moment with temperature. This is done by a scaling law approach, without considering a specific magnetization law and distribution function for the superparamagnetic part, which avoids errors due to the use of an inadequate fit function. This separation is useful because the linear component usually complicates the fit of experimental data and is the reason why the variation of μ_p with temperature cannot be qualitatively inferred in a simple M versus H/T plot. Applying this method to ferritin we conclude that $\langle\mu\rangle$ decreases with temperature and that a distribution cannot be disregarded. In Sec. III, the parameters estimated from applying the scaling law method to ferritin

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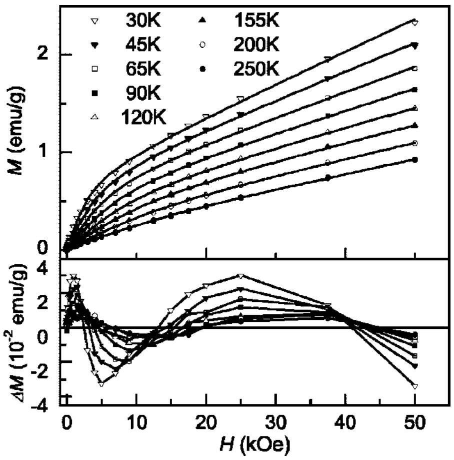

FIG. 1. Magnetization of ferritin as a function of applied field at the indicated temperatures. Solid lines represent fits to the Langevin law [Eq. (2)]. Below: fit residues.

magnetization data are compared with those obtained from fitting the data with distributed and nondistributed Langevin functions. We show that $\langle\mu\rangle$ follows a T^2 temperature dependence, associated with antiferromagnetic magnons. We also investigate the existence of a magnetic moment distribution in ferritin cores not directly related to their size distribution. This system, with narrow size distribution and with negligible interparticle interactions,[18,19] is appropriated to study the influence of disorder in the magnetic moment distribution. In Sec. IV we point out the conclusions.

II. MAGNETIZATION ANALYSIS

A. Magnetization curves fit

The ferritin samples used in these experiments were obtained from Sigma Chemical Company and prepared in powder samples accordingly to Ref. 7. Magnetization was measured with a Quantum Design superconducting quantum interference device (SQUID) magnetometer in magnetic fields up to 50 kOe at several temperatures in the superparamagnetic regime (30–250 K), after field cooling (5 kOe).

Ferritin is a system with low interparticle dipolar fields due to the protein shell that prevents aggregation and to the low particles net magnetic moment. In fact, Allen *et al.*[18] concluded there was a weak interparticle interaction at 5 K and Luis and co-workers[19] have shown negligible differences in ac susceptibility curves of ferritin samples with different concentrations, from diluted to solid samples. In addition, ferritin has a narrow size distribution and therefore one expects a small moment distribution that can be reasonably ignored.[3,7] Within this approximation, we have fitted ferritin magnetization curves with a simple Langevin function [Eq. (1)] and we have obtained an oscillatory residue (Fig. 1) and m_0 and μ_p decreasing and increasing with temperature, respectively (Fig. 2), in accordance with earlier results.[7] A similar systematic oscillatory fit was observed in ferromagnetic[20–22] and antiferromagnetic nanoparticles.[4,6,23] Such behavior of residues and fit parameters is also found when intrinsic distributed data are fitted with the simple Langevin function.[24] The simultaneous increase of μ_p and decrease of m_0 is puzzling and would imply a strong decrease of the particle density N, which has no physical ground.

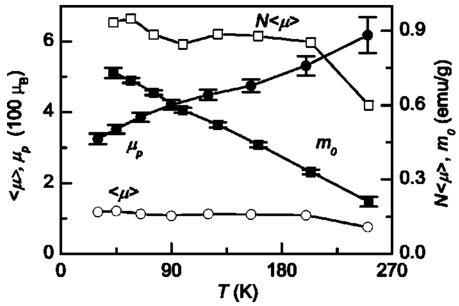

FIG. 2. Parameters μ_p and m_0 (filled symbols) obtained with a nondistributed Langevin function [Eq. (1)] compared with the corresponding parameters $\langle\mu\rangle$ and $N\langle\mu\rangle$, respectively, (open symbols) obtained with a lognormal distributed Langevin function [Eqs. (2) and (3)].

As a second approach we have investigated the existence of a magnetic moment distribution, with the total magnetization expressed by:

$$M(H,T) = N\int_{\mu_{\min}}^{\mu_{\max}} \mu L\left(\frac{\mu H}{kT}\right) f(\mu)d\mu + \chi_{AF}(T)H, \qquad (2)$$

where μ is the particle moment and $f(\mu)$ its normalized lognormal distribution expressed by:

$$f(\mu) = \frac{1}{\mu \cdot s\sqrt{2\pi}} \exp - \frac{\left[\log\left(\frac{\mu}{n}\right)\right]^2}{2s^2}. \qquad (3)$$

The mean particle moment $\langle\mu\rangle$ is equal to $n\sqrt{w}$ with $w=e^{s^2}$, where s^2 is the variance of the normally distributed log(μ). In ideal ferromagnetic superparamagnetic systems, μ is proportional to the volume and the moment distribution arises only due to a volume distribution. In that case it is possible to consider volume distributions instead of moment distribution. However, in real systems, surface disorder, frustration, and spin canting may contribute to moment distributions distinct from volume distributions.[2] This distinction becomes more relevant in the case of antiferromagnetic or ferrimagnetic particles. A much improved fitting is obtained using Eqs. (2) and (3), resulting in residues on the order of data scattering. We note that by using the "random magnetic orientation" function[25–27] together with the lognormal distribution we obtain nonlinear least square values of about 5% higher than using a lognormal distributed Langevin function. As the particle density N obtained in these individual fits is essentially the same for all curves, we performed a global fit imposing the same N for all data. The contrast between $\langle\mu\rangle$ and $N\langle\mu\rangle$ thus obtained and m_0 and μ_p from the nondistributed fit is shown in Fig. 2. Thus, to consider a moment distribution is not just a matter of deriving more accurate parameters but it can drastically change the physical interpretation of the characteristics of superparamagnetic nanoparticles and deserves a more detailed study, as shown in the following sections.

B. Scaling-law method

In the analysis of magnetization curves of antiferromagnetic nanoparticles several problems arise. First, these systems have an antiferromagnetic susceptibility component, which is difficult to separate from the superparamagnetic part, especially if the latter is far from saturation. One important fact is that the departure from saturation depends on the temperature, and thus a high field linear fit gives χ_{AF} in excess and, more drastically, successively distant from the accurate value as the temperature is higher. This is the reason why the method of using linear fits to the asymptotic law and high field regions, suggested by Harris *et al.*,[28] does not avoid the errors introduced by the nondistributed Langevin fit, also noting that m_0 and μ_p decrease and increase with temperature, respectively. At the same time, the variation of χ_{AF} with temperature has not been modeled yet, although a $T^{-1/2}$ variation has been proposed by Gilles *et al.*[25] Second, in antiferromagnetic systems, the superparamagnetic component can be modeled with different functions: the Langevin function, as expressed in Eqs. (1) and (2), a Langevin function with a modified m_0 factor,[28] or the "random magnetic orientation" function derived by Néel.[27] Both Langevin and Néel functions have the same asymptotic behavior but differ in the intermediate field zone, with the latter function saturating at lower fields. Since the use of a distribution function critically changes the parameters temperature variation, in the following paragraphs we present a method to derive qualitative and quantitative information about antiferromagnetic particle parameters variation. This is obtained independently of the magnetization and magnetic moment distribution functions. The method is then applied to ferritin.

In the case of antiferromagnetic nanoparticles with $\langle\mu\rangle$ constant with temperature and with negligible interparticle interactions, the superparamagnetic component scales with H/T, independently of the existence of any moment distribution and of the law that describes the system. The $\chi_{AF}H$ component would scale with H/T only if χ_{AF} obeyed a Curie law. In general, the first derivative of the magnetization with respect to the field multiplied by temperature has a component that collapses in an H/T scale and another component associated with χ_{AF}, in accordance with

$$\frac{\partial M}{\partial H}T = F\left(\frac{H}{T}\right) + \chi_{AF}T, \tag{4}$$

where F is an unknown function of H/T. Thus, if χ_{AF} does not follow a Curie law, a constant difference between the magnetization data obtained at different temperatures will appear in an $(\partial M/\partial H)T$ versus H/T plot, as shown in Fig. 3(a) for ferritin. The increment of $\chi_{AF}T$ in relation to a given temperature T_0 can be evaluated in a $(\partial M/\partial H)T-(\partial M/\partial H)T_0$ representation. At this point, the accuracy of supposing $\langle\mu\rangle$ constant with temperature can be checked by the constancy of $(\partial M/\partial H)T-(\partial M/\partial H)T_0$ with temperature. In the case of native horse-spleen ferritin we chose $T_0=30$ K and we observe a region where $\langle\mu\rangle$ can be considered

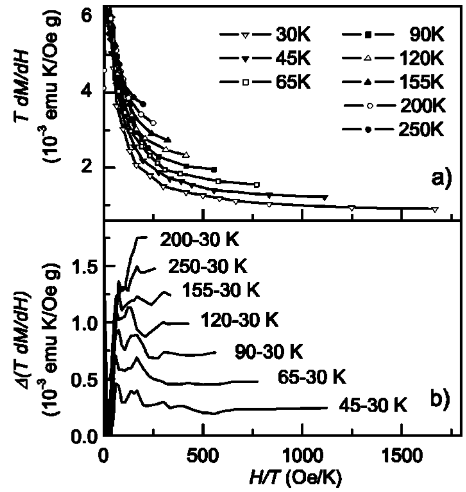

FIG. 3. (a) Representation of $(\partial M/\partial H)T$ as a function of H/T for horse-spleen ferritin and (b) difference between each of the above curves and the $T=30$ K curve as a function of H/T.

constant with H/T [Fig. 3(b)]. $\chi_{AF}(T)$ can then be determined considering the values of $(\partial M/\partial H)T-(\partial M/\partial H)T_0$ in the high H/T region and estimating χ_{AF} at the lowest temperature. This value can be obtained from the extrapolation to zero of $\partial M/\partial H$ as a function of T/H and was estimated as $\chi_{AF}(T_0)=2.6\times10^{-5}$ emu/Oe g. We find that χ_{AF} decreases with temperature [see Fig. 5(a)]. The superparamagnetic component of the magnetization curve of antiferromagnetic particles (M_{SP}) can then be easily obtained by subtracting $\chi_{AF}(T)H$ from the total magnetization and is plotted in Fig. 4(a) for ferritin. As noted, the variation of $(\partial M/\partial H)T-(\partial M/\partial H)T_0$ is not constant with H/T and thus the curves

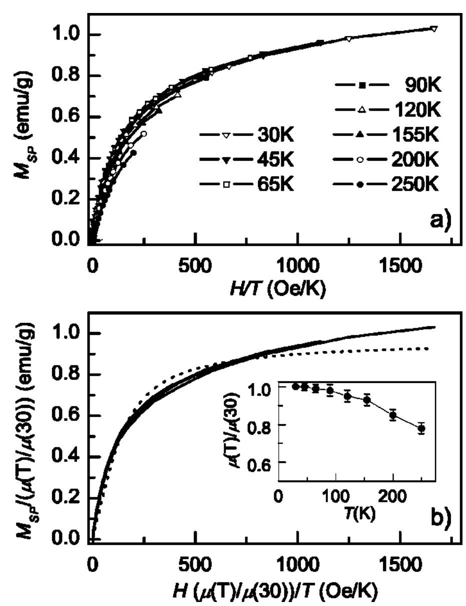

FIG. 4. (a) Ferritin superparamagnetic (saturation) component M_{SP} as a function of H/T; (b) M_{SP} in the scaling plot $M_{SP}/[\mu(T)/\mu(30)]$ vs $H[\mu(T)/\mu(30)]/T$. The nondistributed Langevin fit is shown as a dotted line. The inset shows the relative $\langle\mu\rangle$ temperature variation.

do not superimpose on H/T, meaning that $\langle\mu\rangle$ varies with temperature. An important observation is that the curves saturate successively at higher H/T values as the temperature of measurement is higher. We can therefore conclude that in ferritin $\langle\mu\rangle$ decreases with temperature, without any assumption of a particular function or distribution. In a general case, if the M_{SP} curves scale, a single fit to all temperatures can be performed and several laws and distribution functions can be tested, avoiding χ_{AF} and knowing beforehand that $\langle\mu\rangle$ and N are constant with temperature. In antiferromagnetic systems where $\langle\mu\rangle$ is found to be temperature dependent, as in our ferritin samples, we can still distinguish between situations where a μ distribution can or cannot be ignored. On the condition that the distribution is narrow or the variation of μ with temperature is small compared with the distribution deviation, there is a scaling factor for each curve such that dividing M_{SP} and multiplying H/T by this same factor scales all curves. In other words, it is possible to find a $M_{SP}/\mu(T)$ versus $H\mu(T)/T$ scaling plot. In order to find this scaling plot, the lower temperature curve can be set as a reference and the ratio $\mu(T)/\mu(T_0)$ derived. In our ferritin magnetization curves there are no such scale factors and thus a distribution function cannot be ignored. However, ferritin approaches the case where the variation of $\langle\mu\rangle$ with temperature is small compared with the distribution deviation. Accordingly, a good scaling [Fig. 4(b)] and an estimation of the $\mu(T)/\mu(30)$ ratio [inset of Fig. 4(b)] are obtained. The $\langle\mu\rangle$ decrease ratio is 0.78 ± 0.03 when the temperature increases from 30 to 250 K.

In summary, without knowing the particular distribution function or the individual particle magnetization law, this method gives information about the χ_{AF} and μ temperature dependence. The absolute scale of χ_{AF} and $\langle\mu(T)\rangle$ are determined by χ_{AF} and μ at the reference temperature T_0. The subsequent ferritin magnetization curves analysis is therefore enlightened by the information derived here, namely that the distribution function cannot be ignored and that $\mu(T)$ decreases with temperature.

III. DISCUSSION

An agreement between the χ_{AF} and $\langle\mu(T)\rangle$ variation obtained with the fit using a distributed Langevin function and the scale method (the latter giving a smoother variation at higher temperatures) is observed (Fig. 5). A decrease of the average magnetic moment with temperature was already found by Gilles *et al.* for artificially reconstructed ferritin cores, using a "random magnetic orientation" function and a lognormal distribution function, imposing the size distribution and a power-law relation between magnetic moment and volume.[25] Therefore, based on the scaling and on the distributed fits we conclude that $\langle\mu\rangle$ decreases with temperature and that ignoring the existence of a moment distribution is the cause of the artificial increase of $\langle\mu\rangle$ and decrease of m_0 previously reported in ferritin.[3,7] The decrease of m_0 was also found by several authors in other anitiferromagnetic particles systems as ferrihydrite,[4,5] ferrihydrite doped with Ni, Mo, and Ir,[29] and NiO,[6,7] and was tentatively associated with a

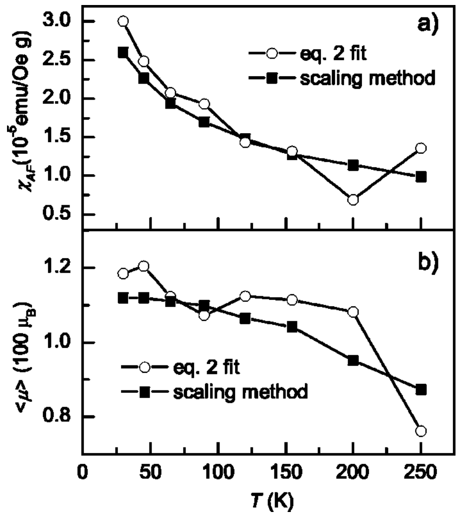

FIG. 5. χ_{AF} (above) and $\langle\mu\rangle$ (below) obtained with a lognormal function in Eq. (2) compared with the values obtained with the scaling method.

surface moment intrinsic behavior. Our results show that care must be taken to ensure that such a m_0 variation has physical meaning and does not come from ignoring a magnetic moment distribution. At the same time, the increase of μ_p based on a nondistributed analysis was also observed in artificial ferritin with different core mean sizes[28] and ferrihydrite particles.[4,5] A closer look reveals that the strongest variations reported take place in powder ferrihydrite samples[4] and in the smaller artificial ferritins,[28] where a wider volume and thus μ distribution are likely to occur. Such an apparent temperature assisted onset of magnetic moments was associated with weaker exchange, strong radial anisotropies, frustration, multiple sublattices (Ref. 28 and references therein), and interparticle interactions.[4] In addition, recent work interprets this anomalous behavior as dynamic thermoinduced magnetization.[9] Despite the possible contribution of all these features in the referred systems, we show that the existence of a μ distribution leads to an analogous μ_p artificial temperature variation that must be carefully analyzed (see also Ref. 24). Thus, the structure information,[4–7] the spin arrangements,[7] the thermoinduced magnetization,[9] and deviations from the Curie law[8] derived based on that increase raise severe doubts.

Since the mean magnetic moment results from the uncompensated/canted moments of the antiferromagnetic configuration, we may expect that, in a first approximation, $\langle\mu(T)\rangle$ follows the bulk antiferromagnetic magnons law[25] $\langle\mu(0)\rangle(1-\alpha T^2)$ (Fig. 6). Extrapolating to $\langle\mu\rangle=0$ one obtains an estimation of the Néel temperature, $T_N\approx500$ K, in accordance with the value derived in Ref. 25. Recent neutron diffraction investigations performed on 2.8 nm diam ferrihydrite powder particles show that $T_N=330\pm30$ K.[30] The difference between this value and our T_N estimation may be due to the difference in the particles size (our ferritin sample has a diameter of up to 5 nm) since it is likely that T_N increases with size. However, 500 K is probably an overestimation of T_N, because the T^2 law is not expected to hold up to such high temperature. The value of $\langle\mu(30\text{ K})\rangle=120$ μ_B

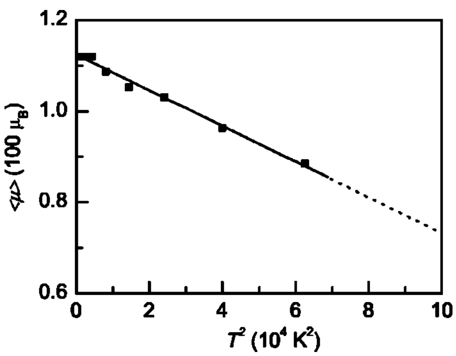

FIG. 6. Plot of $\langle\mu\rangle$ against T^2, where the agreement with an antiferromagnetic magnons law (solid line) can be observed.

obtained with the distributed Langevin fit is about five times lower than the value obtained using a nondistributed Langevin function [Eq. (2)] and about 0.8 of the value obtained in Ref. 25. This value corresponds to a mean number of fully uncompensated Fe^{3+} ions N_{un} of 23. The number of Fe^{3+} ions involved in the superparamagnetism is obviously greater, as a range between fully compensated and fully uncompensated configurations is expected. Since the mean horse-spleen ferritin core has a total number of Fe ions N_t of about 2000–3000,[17] N_{un} is on the order of N_t^p with $1/2 < p < 1/3$, which suggests that the uncompensated spins are not only at the surface but also randomly distributed through the volume. The values of s obtained with the fit procedure vary from 0.9 at 30 K to 1.3 at 65 K and to 1.0 at 250 K. These values are about ten times greater than the typical value of the ferritin diameter distribution.[17,25] Such a difference is not compatible with the assumption that the uncompensated moments distribution is just a consequence of volume distribution and that the number of uncompensated moments is a fixed power p of the volume. In fact, $s=1$ implies the existence of particles with a maximum number of fully uncompensated Fe^{3+} ions of about 100 ions, which would mean 10 000 total ions with $p=1/2$, while the ferritin maximum Fe loading is about 5000 ions. Thus, if the Langevin or the "random magnetic orientation" suitably describes the uncompensated moment of ferritin cores, we are led to the conclusion that an important magnetic moment distribution exists even in a case where the size distribution is of minor importance. This emphasizes the existence of particles with approximately the same size but different degrees of inner or surface structure/magnetic disorder.

IV. CONCLUSIONS

In this paper, we have presented a method based on scaling laws to obtain the variation of the antiferromagnetic susceptibility χ_{AF} and mean uncompensated moment $\langle\mu\rangle$ with temperature from the magnetization curves of antiferromagnetic nanoparticles. If $\langle\mu\rangle$ depends on the temperature, we can also determine whether the system can or cannot be described without a distribution function. Applying this method to ferritin we found that $\langle\mu\rangle$ decreases with temperature and that a distribution function cannot be ignored. This is in contrast with some previous results, where $\langle\mu\rangle$ was found to increase with temperature after fitting the magnetization curves with a nondistributed Langevin function. This disagreement arises from ignoring a moment distribution in the analysis of the magnetization curves and emphasizes the need to evaluate the effect of distributions in ferritin and in other antiferromagnetic systems as ferrihydrite and NiO. The fit of ferritin magnetization curves with Langevin and log-normal functions yielded parameters on the order of those estimated with the scaling method and confirmed the decrease of $\langle\mu\rangle$ with temperature. The moment distribution thus obtained is not simply related to the narrow size distribution characteristic of ferritin. Therefore we are led to conclude that the existence of important intraparticle magnetic disorder is the source of moment distribution.

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*Corresponding author. Electronic address: nunojoao@fis.ua.pt

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