1. Introduction

The colossal magnetoresistance (CMR) and the magneto-caloric (MC) effects discovered in doped rare-earth manganite perovskites (denoted as manganites) \( \text{Re}_{1-x}M_x\text{MnO}_3 \) (\( \text{Re}=\text{La, Pr, Nd} \ldots \); \( M=\text{Ca, Sr, Ba} \ldots \)) around the ferromagnetic (FM)-paramagnetic (PM) phase transition have attracted a great deal of interest due to their potential applications as field sensors, memory recording, magnetic reading heads, magnetic refrigeration, and novel electronic materials [1–6]. Earlier studies have also shown that the magnetic and transport properties of manganites depend on the double-exchange interaction of \( \text{Mn}^{3+} \) and \( \text{Mn}^{4+} \) ions [7], the Jahn-Teller effect [8], the phase separation combined
with percolation [9], as well as the oxygen deficiency [10]. However, the origin of the observed phenomena is still not fully understood. In the recent years, scientists have concentrated on the critical behaviour around the FM-PM phase transition of manganites in order to better understand the nature of the magnetic interactions and to derive the critical exponents (β, γ, and δ). The critical behaviour in the double-exchange interaction was described in a theoretical work by the long-range mean-field theory (MFT) with β = 0.5 and γ = 1.0 [11]. In 2000, Motome and Furulawa [12] used the computational technology to study the critical properties for some manganites. They suggested that their FM order is short-range one, which should belong to the 3D-Heisenberg model. On the contrary, Shin et al. [13] reported β = 0.387 and γ = 1.166 for La0.7Sr0.3MnO3. These results are in good agreement with those earlier reported by Ghosh et al. [14]. Magnetization isotherms D8 AXS, Discover) and by using the Williamson-Hall method (D8 AXS, Discover) and by using the Williamson-Hall method [15]. Whereas, a relatively high value of β determined in some manganites, such as Nd0.6Sr0.4MnO3 (β = 0.51) [16], La0.7Ca0.3MnO3 (β = 0.491) and La0.7Ba0.3MnO3 (β = 0.493) [17], and Pr0.55Sr0.45MnO3 (β = 0.462) [18] is very close to that of the MFT. On the contrary, Shin et al. [19] reported a very low value of β obtained in La0.7Ca0.3MnO3 (β = 0.14), which suggests that the magnetic phase transition in this material should be the first-order. Therefore, in view of the varied values of the critical exponents in manganites, the theoretical models including the mean-field model [11], the 3D-Heisenberg model [12], the 3D-Ising model [20]; and the tricritical mean-field model [21] were used to explain their critical properties.

In a previous report, Thanh et al. [22] have used the modified Arrott plot method to determine the critical exponents β, γ, and δ for Pr0.6Sr0.4MnO3 (PSMO) nanocrystals (NCs). They pointed out an existence of the long-range FM order in these compounds. However, in this work, using the MFT [11], the Kouvel-Fisher method [23], and M (H, T) data, the critical properties around the FM-PM transition of these PSMO NCs have been investigated in more detailed. We have also estimated their spontaneous magnetization values based on the magnetic entropy change data. Thereby, the value of exponent β can be predicted. Additionally, the range of the exchange interaction (σ), the correlation length critical exponent (ν), the heat capacity critical exponent (α), as well as the reduced temperature (ε = (T - Tc)/Tc) dependences of the effective exponents βeff (ε) and γeff (ε) for PSMO NCs have been also mentioned.

2. Experimental details

In the present work, we used samples that were taken from the similar batches investigated in a previous work [22]. Three samples of PSMO NCs with different crystallite sizes (D = 39, 59, and 88 nm) were prepared by a combination of the solid state reaction and the mechanical ball milling methods with various milling times (tm = 30, 20, and 10 min). Herein, the bulk samples were prepared by the solid state reaction method with a sintering process at 1300 °C for 48 h in air, using high-purity powders (99.9%) Pr2O3, SrCO3, and Mn. Next, these bulks were ground into powder, and then were used for the mechanical ball milling to make PSMO NCs. Detailed descriptions for this synthesis can be found elsewhere [22]. The structure analyses and the average crystallite size were carried out on an x-ray diffractometer (Bruker D8 AXS, Discover) and by using the Williamson-Hall method [24]. Magnetization isotherms M (H) were measured on a superconducting quantum interference device (SQUID) magnetometer in the range of H = 0–30 kOe and using a warming mode with a temperature interval of ΔT = 2K in the vicinity of Tc. The applied magnetic field (Happ) has been corrected by a demagnetization factor (k) that has been determined by a standard procedure from M (Happ) data in the low-field linear response regime at low temperature (H = Happ - kM) [25].

3. Results and discussion

As shown in figure 1(a), room-temperature x-ray diffraction (XRD) patterns proved a single phase of Pr0.6Sr0.4MnO3, belonging to the orthorhombic structure (space group: Pbnm) [25]. Based on the XRD data, the average crystallite size (D) has been calculated through the Williamson-Hall method [24]. Figure 1(b) shows experimental data fitted to the Williamson-Hall relation for samples to estimate the (D) values. Detailed descriptions for this content can be found elsewhere [22, 24]. As a result, (D) values are obtained to be 39, 59, and 88 nm corresponding to tm = 30, 20, and 10 min.

![Figure 1.](image-url)
In an earlier report [22], we pointed out that PSMO NCs are soft magnetic materials. They undergo a second-order magnetic phase transition corresponding to $T_C = 286 - 291 \text{K}$ for $\langle D \rangle = 39 - 88 \text{ nm}$, respectively. Based on the magnetic-field dependences of magnetization $M(H)$ measured at various temperatures [22], the temperature dependences of magnetic entropy change $\Delta S_m(T)$ can be investigated via the thermodynamic Maxwell relationship [26]

$$\Delta S_m(T,H) = S_m(T,H) - S_m(T,0) = \frac{\partial S}{\partial H} dH = \frac{\partial M}{\partial T} dH.$$  

(1)

According to the thermodynamic theory, the relation between the heat capacity $C_{P,H}$ and the magnetic entropy change of a material can be given as [26]

$$\Delta S_m(T,H) = \int_0^T \frac{C_P(T,H) - C_P(T,0)}{T} dT.$$  

(2)

Therefore, the specific heat change $\Delta C_{P,H}$ associated with a magnetic field change $\Delta H$ from 0 to $H$ can be defined by

$$\Delta C_P(T,H) = C_P(T,H) - C_P(T,0) = T \frac{\partial \Delta S_m}{\partial T}.$$  

(3)

As shown in figures 2(a)–(c), all samples exhibit a MC effect around the magnetic phase transition. A maximum value of the magnetic entropy change appears on $\Delta S_m(T)$ curve. For each sample, this maximum value increases with increasing applied field. Based on $\Delta S_m(T)$ data, $\Delta C_{P,H}(T)$ data of PSMO NCs under several applied magnetic field changes $\Delta H = 5 - 30 \text{ kOe}$ has been calculated through equation (3) and performed in figures 2(d)–(f). All $\Delta C_{P,H}(T)$ curves exhibit anomaly near $T_C$ due to the FM-PM transition. The value of $\Delta C_{P,H}$ strongly changes from the negative value to the positive one as temperature increases. With an enhancement of applied field, the $|\Delta C_P|$ value monotonically increases. For the applied field change of 10 kOe, the $\Delta C_P^{\text{max}} / \Delta C_P^{\text{min}}$ value is found to be about $30 / -20 \text{ } \text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$. Besides, both the changes of magnetic entropy and the specific heat decrease gradually with decreasing crystallite size $\langle D \rangle$ from 88 nm to 39 nm. These reductions could be assigned to the enhancement of the nonmagnetic layer or spin disorder on the particle surface when particle size decreases [22].

To get more information about the magnetic interactions in PSMO NCs, we used the MFT to determine the temperature dependences of spontaneous magnetization ($M_S$). According to the MFT, in the FM region, the magnetic entropy change of a second-order material can be performed as a linear function of the magnetization ($\Delta S_m = f \left[ M^2 \right]$) [27]. Therefore, the value of the spontaneous magnetization at temperature $T$ below $T_C$ can be determined through the intersection of the straight line $\Delta S_m = f \left[ M^2 \right]$ with the $M^2$ axis. Figures 3(a)–(c) perform $-\Delta S_m = f \left[ M^2 \right]$ data at different temperatures in the
FM region of PSMO NCs. Clearly, $-\Delta S_m = f \{M^2(T)\}$ data at different temperatures exhibit a set of linear dependences. However, these straight lines are not absolutely parallel, and their divergence increases when crystallite size decreases. This suggests that the magnetic interactions in PSMO NCs do not fully belong to the MFT.

Figures 3(d)–(f) show the $M_S(T)$ data (symbols) for PSMO NCs, which are determined from the intersections of the linear extrapolation lines of $-\Delta S_m = f \{M^2(T)\}$ at several temperatures in the FM region with the $M^2$ axis. With increasing temperature, the spontaneous magnetization of PSMO NCs monotonously decreases. Based on the power law of the spontaneous magnetization in the temperature region near the FM-PM phase transition \[11\]

$$M_S(T) = M_0 \left(\frac{T_C - T}{T_C}\right)^\beta.$$  \(4\)

To determine the value of critical exponent $\beta$, which describes how the order moment grows below $T_C$ of PSMO NCs, we fitted their $M_S(T)$ data to equation (4), see the solid lines in figures 3(d)–(f). By this way, we obtained the values of $T_C$ ($T_C = 292.4 \pm 0.1, 290.6 \pm 0.1,$ and $290.1 \pm 0.1$ for $\langle D \rangle = 88, 59,$ and $39$ nm, respectively) and $\beta$ ($\beta = 0.508 \pm 0.011, 0.489 \pm 0.009,$ and $0.486 \pm 0.012$ for $\langle D \rangle = 88, 59,$ and $39$ nm, respectively). We can see that the value of critical exponent $\beta$ obtained for PSMO NCs is very close to that of the MFT ($\beta = 0.5$ \[11\]). It means that FM order in the samples is the nearest long-range FM order. However, the reduction in value of $\beta$ in the samples with smaller crystallite sizes suggests a small deviation from the MFT.

To evaluate more accurately the value of the critical parameters, we used the Kouvel-Fisher method, which can independently determine the value of the critical exponents $\beta$ and $\gamma$ through the Kouvel-Fisher plots \[28\]. According to this method, the relationship between the spontaneous magnetization $M_S$, the initial susceptibility $\chi$, the critical exponents $\beta$ and $\gamma$, and temperature $T$ can be defined as \[23\]

$$Y_1(T) = \frac{M_S(T)}{dM_S(T)/dT} = \left(\frac{T - T_C}{\beta}\right),$$ \[5\]

$$Y_2(T) = \frac{\chi_0^{-1}(T)}{d\chi_0^{-1}(T)/dT} = \left(\frac{T - T_C}{\gamma}\right).$$ \[6\]

Because $M_S(T)$ and $\chi_0^{-1}(T)$ around Curie temperature of the second-order phase transition obey the power laws with the exponents $\beta$ and $\gamma$, both $Y_1(T)$ and $Y_2(T)$ plots should...
be straight lines in the critical region, and the slopes of $Y_1(T)$ and $Y_2(T)$ lines give the values of $\beta$ and $\gamma$ [28]. Based on $M(H)$ data measured at different temperatures in the critical region, $Y_1(T)$ and $Y_2(T)$ data have been determined and performed in figures 4(a)–(c). Herein, the straight lines are linear fits $Y_1(T)$ and $Y_2(T)$ data to equations (5) and (6) for PSMO NCs. The critical parameters of the samples are found to be $\beta = 0.492 \pm 0.009$, $\gamma = 1.019 \pm 0.008$, and $T_C = 290.5 \pm 0.1$ K for $\langle D \rangle = 88$ nm; $\beta = 0.488 \pm 0.006$, $\gamma = 1.056 \pm 0.010$, and $T_C = 288.7 \pm 0.1$ K for $\langle D \rangle = 59$ nm; and $\beta = 0.461 \pm 0.010$, $\gamma = 1.062 \pm 0.005$, and $T_C = 285.3 \pm 0.1$ K for $\langle D \rangle = 39$ nm.

For the third critical exponent $\delta$ (associated with the critical isotherm), it can be calculated by using the obtained values of $\beta$ and $\gamma$ via the Widom scaling relation [11]

$$\delta = 1 + \frac{\gamma}{\beta}. \quad (7)$$

By this way, $\delta$ value for PSMO NCs is found to be 3.071, 3.164, and 3.304 for $\langle D \rangle = 88, 59,$ and 39 nm, respectively. To check the reliability of the obtained critical parameters, we have used the scaling function of magnetization data [29]

$$\frac{H}{M^\delta} = h\left(\frac{\varepsilon}{M^{1/\beta}}\right), \quad (8)$$

where $h$ and $\varepsilon = (T - T_C)/T_C$ are the scaling function and the reduced temperature, respectively. Equation (8) implies that the scaled $M/H^{1/\delta}$ versus $\varepsilon/H^{1/(\beta + \gamma)}$ (with $\Delta = \beta\gamma = \beta + \gamma$ is the gap exponent) will collapse onto a universal curve with all experimental data points if we used the appropriate values of the critical parameters [29]. Thus, in this work, the values of the critical parameters ($\beta$, $\gamma$, $\delta$, and $T_C$) obtained from the Kouvel-Fisher method as mentioned above have been used to construct $M/H^{1/\delta}$ versus $\varepsilon/H^{1/\Delta}$ and performed in figures 4(d)–(f). Clearly, most of $M(H,T)$ data points in the critical region of PSMO NCs fall onto a universal curve. This result confirms that the obtained value of $\beta$, $\gamma$, $\delta$, and $T_C$ are appropriate.

In comparison with the critical exponents of the theoretical models (the mean-field model $\beta = 0.5$ and $\gamma = 1.0$ [11], the 3D-Heisenberg model $\beta = 0.365$ and $\gamma = 1.336$ [12], the 3D-Ising model $\beta = 0.325$ and $\gamma = 1.241$ [20], and the tricritical mean-field model $\beta = 0.25$ and $\gamma = 1.0$ [21]) the values of the exponents obtained for PSMO NCs are quite close to those expected for the MFT. This proves the existence of a long-range FM order in the samples. However, the values of $\beta$ obtained for the samples are smaller than 0.5, suggesting an existence of the magnetic inhomogeneities, which can be explained by the contribution of the nonmagnetic layer and/or spin disorder on the surface of the nanoparticles, which leads to the weakening of the double-exchange interaction strength [30].
Zhang and co-workers [31] pointed out that a small deviation of the critical exponents from the values of the theoretical models often appears in a magnetic system that is governed by various competing coupling and/or disorders. Therefore, the critical exponents belonging to any universality class in the asymptotic region should be investigated. To further testify the convergence of the critical exponents for PSMO NCs, we calculated their effective exponents $\beta_{\text{eff}}$ and $\gamma_{\text{eff}}$ via the relations [31]

$$\beta_{\text{eff}}(\varepsilon) = \frac{d[\ln M_3(\varepsilon)]}{d[\ln \varepsilon]},$$

$$\gamma_{\text{eff}}(\varepsilon) = \frac{d[\ln \chi_0^{-1}(\varepsilon)]}{d[\ln \varepsilon]}.$$  

Using equations (9) and (10), the reduced temperature dependences of the effective exponents $\beta_{\text{eff}}(\varepsilon)$ and $\gamma_{\text{eff}}(\varepsilon)$ of PSMO NCs are calculated and presented in figure 5. This exhibits a non-monotonicity of $\beta_{\text{eff}}$ and $\gamma_{\text{eff}}$ of PSMO NCs do not match any theoretical universality classes. However, when temperature is approaching $T_C (\varepsilon \to 0)$, the values of $\beta_{\text{eff}}$ and $\gamma_{\text{eff}}$ for $\langle D \rangle = 88$ and $59$ nm approach the values expected for the MFT (figures 5(a) and (b)), whereas $\beta_{\text{eff}}$ and $\gamma_{\text{eff}}$ of $\langle D \rangle = 39$ nm shift to the values located in between those of the MFT and the 3D-Heisenberg or 3D-Ising models (figure 5(c)). The non-monotonicity of $\beta_{\text{eff}}$ and $\gamma_{\text{eff}}$ versus $\varepsilon$ observed for our samples agrees with earlier report for Pr$_{0.5}$Sr$_{0.5}$MnO$_3$ compound by Pramanic and Banerjee [32], who suggested that the non-monotonic changes of $\beta_{\text{eff}}$ and $\gamma_{\text{eff}}$ were attributed to magnetic disorders in the samples. In a recent report, Zhang et al [31] commented that the non-monotonic changes of $\beta_{\text{eff}}(\varepsilon)$ and $\gamma_{\text{eff}}(\varepsilon)$ are an intrinsic property because the critical exponents of a homogeneous magnetic system are independent of the microscopic details, which is due to a divergence of the correlation length in the asymptotic critical region.

According to Fisher and co-workers [33], the universality class of a homogeneous magnetic system exhibiting the second-order phase transition depends on the range of the exchange interactions ($\sigma$), which appears in the exchange integral $J(r) \sim r^{-(d+\sigma)}$, where $r$ is the distance, $d$ is the spatial dimensionality. For homogeneous magnets with dimensionality $d$ and spin $n$, the range of the exchange interactions $\sigma$ can be calculated via the relation [31]

$$\gamma = 1 + 4\frac{(n + 2)}{d(n + 8)} \Delta \sigma + \frac{8(n - 4)(n + 2)}{d^2(n + 8)^2} \left[ 1 + \frac{2G(\frac{d}{4})}{(n - 4)(n + 8)} \right] \Delta \sigma^2,$$

where $\Delta \sigma = \sigma - 1/2 d$ and $G(1/2 d) = 3 - 1/4 (1/2 d)^2$. For three-dimensional materials ($d = 3$), the exchange integral can be defined as $J(r) \sim r^{-(3+\sigma)}$. The Heisenberg model is valid for the $3D$ isotropic ferromagnet when $\sigma \geq 2$, where the exchange integral $J(r)$ decreases faster than $r^{-3}$. Meanwhile, the MFT is valid for $\sigma \leq 3/2$. It means that the exchange integral $J(r)$ decreases more slowly than $r^{-4.5}$. And in the range $3/2 < \sigma < 2$, the system follows different class depending on the value of $\sigma$ [33]. For our case, the value of $\gamma$ obtained from the Kouvel-Fisher method as mentioned above has been used to calculate the range of the exchange interaction $\sigma$ for PSMO NCs. Thus $\sigma$ value of the samples whenever $d = 3$ and $n > 1$ is found to be $1.542, 1.589$, and $1.594$ for $\langle D \rangle = 88, 59$, and $39$ nm, respectively. Clearly, these values are very close to $\sigma = 3/2$ that is expected for the MFT. This result suggests that the exchange interactions in PSMO NCs obey the MFT, which is valid for the long-range FM orders.

Additionally, the remaining critical exponents including the correlation length ($\nu$) and the heat capacity ($\alpha$) critical exponents have also been calculated via the below relations

\[\text{Figure 5.} \text{ The reduced temperature dependences of the effective exponents } \beta_{\text{eff}}(\varepsilon) \text{ and } \gamma_{\text{eff}}(\varepsilon) \text{ in the asymptotic region.}\]
\[ \nu = \frac{\gamma}{\sigma}, \quad (12) \]
\[ \alpha = 2 - \nu d. \quad (13) \]

Using equations (12) and (13), the values of \( \nu \) and \( \alpha \) are found to be \( \nu = 0.659, 0.665, \) and \( 0.666 \) and \( \alpha = 0.023, 0.005, \) and \( 0.002 \) for \( \langle D \rangle = 88, 59, \) and \( 39 \text{nm} \), respectively. These results seem to be in agreement with the predicted one for the MFT \( (\alpha = 0) \) [34]. The self-consistency of the critical exponents determined for the samples proves that their values are reliable and unambiguous.

4. Conclusion

In summary, we have prepared the nanosized \( \text{Pr}_{0.6}\text{Sr}_{0.4}\text{MnO}_3 \) polycrystalline samples with \( \langle D \rangle = 39 – 88 \text{ nm} \) by a combination of the solid state reaction and the mechanical ball milling methods during \( \tau = 10 – 30 \text{ min} \). Based on the magnetic entropy change data, the spontaneous magnetization of samples can be determined. By this way, the exponent \( \beta \) can be easily estimated. The analysis based on the Kouvel-Fisher method shows that the samples undergo the second-order phase transition with the values of the critical exponents \( \beta = 0.461 – 0.492, \gamma = 1.019 – 1.062, \) and \( \delta = 3.071 – 3.164 \) approximating the values of the MFT. This suggests that the FM order in the samples is long-range order. Besides, the reduction of the exponent \( \beta \) in the samples with smaller crystallite sizes has also been observed \( (\beta = 0.492 \) and 0.461 for \( \langle D \rangle = 88 \) and 39 nm, respectively). This proves an existence of the magnetic inhomogeneities. We have pointed out the non-monotonicness of \( \beta_{\text{eff}} (\varepsilon) \) and \( \gamma_{\text{eff}} (\varepsilon) \) in the asymptotic region of all the samples. These non-monotonic changes are ascribed to magnetic disorders in the samples.

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