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Synthesis and Electrical Characterization of Spinel $M\mathrm{Fe_2O_4}$ (M = Ni, Co, Fe) Magnetic Nanoparticles

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In this investigation, we delve into the precise synthesis of magnetic nanoparticles with a particular emphasis on the $\mathrm{NiFe_2O_4}$ and $\mathrm{CoFe_2O_4}$ ferrites, and the $\mathrm{FeFe_2O_4}$ magnetite, employing a meticulously controlled chemical-synthesis methodology to ensure reproducibility and optimal material properties. To verify the chemical makeup and purity of these synthesized nanoparticles, we conduct the detailed characterization through energy-dispersive x-ray spectroscopy (EDX), which provides quantitative insights into their elemental composition. Leveraging these well-characterized nanoparticles as the foundational components, we formulate a series of magnetic fluids at diverse concentration levels, tailoring their rheological and magnetic behaviours for potential applications in fields such as biomedicine, electronics, and environmental remediation. Additionally, to assess comprehensively the thermoelectrical performance of these magnetic fluids, we perform systematic measurements of their electrical conductivity over an extensive temperature spectrum, thereby, elucidating how thermal variations influence their conductive characteristics and overall functionality under varying operational environments.

Key words: magnetic nanoparticles, spinel ferrite nanoparticles, electrical conductivity, thermoelectrical properties, concentration effects, magnetic fluid, nanoparticles' dispersion.

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У цьому дослідженні ми розглядаємо прецизійну синтезу магнетних наночастинок із особливим акцентом на феритах Ni_2Fe_2O, $CoFe_2O_4$ та магнетиті $FeFe_2O_4$, використовуючи ретельно контрольовану методологію хемічної синтези для забезпечення відтворюваности й оптимальних властивостей матеріялу. Для підтвердження хемічного складу та чистоти синтезованих наночастинок було проведено детальну характеризацію за допомогою енергодисперсійної рентґенівської спектроскопії (EDX), що забезпечило кількісну оцінку їхнього елементного складу. Використовуючи ці добре охарактеризовані наночастинки в якості базових компонентів, було створено серію магнетних рідин із різними концентраційними рівнями, що уможливило цілеспрямовано модифікувати їхні реологічні та магнетні властивості для потенційного застосування в таких галузях, як біомедицина, електроніка й послаблення впливу на довкілля. Крім того, з метою комплексної оцінки термоелектричних характеристик цих магнетних рідин було проведено систематичні міряння їхньої електропровідности в широкому температурному діяпазоні, що дало змогу з'ясувати вплив температурних змін на їхні провідні властивості та загальну функціональність у різних умовах експлуатації.

Ключові слова: магнетні наночастинки, наночастинки шпінельного фериту, електропровідність, термоелектричні властивості, концентраційні ефекти, магнетна рідина, дисперсія наночастинок.

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1. INTRODUCTION

The rapid advancement of science and technology in the 21st century has posed significant environmental challenges, necessitating innovative solutions from materials science and physics. Magnetic fluids, or ferrofluids, have emerged as versatile nanomaterials with promising potential to address these issues through their unique magneto-responsive properties [1, 7]. These colloidal suspensions, comprising magnetic nanoparticles dispersed in a carrier liquid with stabilizing surfactants, exhibit tuneable magnetic, electrical, and rheological behaviours, enabling applications in aerospace engineering, electronics, mechanical systems, biomedicine, and environmental remediation such as water and oil purification [5, 6]. This broad applicability has garnered substantial interest from the scientific community, driving research into their synthesis, characterization, and property optimization.

Magnetic fluids typically consist of magnetic nanoparticles dispersed in a liquid carrier medium and stabilized by surface-active agents. Among these components, magnetic nanoparticles are the primary determinants of the materials' performance [4, 9]. While the chemical composition governs the general quality of the fluid, the size, morphology, and crystal structure of the nanoparticles critically in-

fluence their fundamental physical, chemical, and functional properties. In particular, nanoparticles with the general formula MFe$_2$O$_4$ (M = Ni, Co, Fe) belong to the family of spinel ferrites, which are known for their remarkable magnetic and electrical characteristics [1, 2, 6]. However, the microstructural features and the mechanisms underlying the variations in magnetization and electrical behaviour in fluids containing mixed-metal ferrite nanoparticles remain insufficiently understood [5].

Despite extensive experimental and theoretical investigations, no unified framework describing the internal structure–property relationships of MFe$_2$O$_4$-based magnetic fluids has been established. Furthermore, the origins of variability in their electrical properties, especially as a function of composition, concentration, and temperature, have not been comprehensively explored. In this study, magnetic nanoparticles of MFe$_2$O$_4$ (M = Ni, Co, Fe) were synthesized *via* a chemical condensation method [11, 12]. The chemical composition of the nanoparticles was characterized using Energy-Dispersive X-Ray Spectroscopy (EDX). Magnetic fluids with varying nanoparticle concentrations, prepared using water as the base medium, were evaluated for their electrical conductivity across a range of temperatures. The findings of this work aim to contribute to the understanding of composition–structure–property relationships in spinel ferrite-based magnetic fluids, thereby, facilitating their targeted design for advanced engineering and environmental applications [4, 7].

2. EXPERIMENTAL DETAILS

2.1. Materials

Ferric chloride hexahydrate ($FeCl_3 \cdot 6H_2O$, ≥99%), ferrous chloride tetrahydrate ($FeCl_2 \cdot 4H_2O$, ≥99%), aqueous ammonia (NH_3(aq), 25%), oleic acid ($C_{18}H_{34}O_2$, ≥99%), citric acid ($C_6H_8O_7$, ≥99.5%), and acetone (C_3H_6O, ≥99.5%) were used as received. Deionized water (resistivity ≥ 18.2 MΩ·cm) was used in all preparations.

2.2 Synthesis of magnetite ($FeFe_2O_4$) nanoparticles

$FeFe_2O_4$ nanoparticles were prepared by chemical co-precipitation. Briefly, 2.0 g $FeCl_2 \cdot 4H_2O$ and 5.6 g $FeCl_3 \cdot 6H_2O$ (molar ratio FeI^+:FeI^+ = = 1:2) were dissolved in 100 mL deionized water and heated to 96°C under vigorous stirring. A total of 20 mL NH_4OH was added dropwise *via* syringe; the suspension was maintained at 96°C for 55 min. Then, 4.0 g citric acid (in 20 mL water) and 20 mL oleic acid were added sequentially. The mixture was heated to 100°C and stirred for an addi-

tional 90 min.

A small aliquot was diluted twofold and exposed to a constant magnetic field (0.35 T) to confirm magnetic response; the colloid remained stable in zero field. Excess unbound acids were removed by standing for 24 h followed by filtration. The precipitate was dried at room temperature for 48 h to yield $FeFe_2O_4$ nanopowder.

2.3 Synthesis of $NiFe_2O_4$ and $CoFe_2O_4$ nanoparticles

$NiFe_2O_4$ and $CoFe_2O_4$ nanoparticles were synthesised analogously by substituting the corresponding divalent metal salts for FeI^+ while maintaining the overall FeI^+:MI^+ stoichiometry (M = Ni or Co). Post-addition, the suspensions were aged at 100°C for 90 min, purified, and dried as above to obtain the respective ferrite nanopowders.

2.4 Reaction chemistry and stoichiometry

The co-precipitation proceeds *via* hydroxide formation, followed by in-situ dehydration/condensation to the spinel ferrite under hydrothermal-like ageing (≅ 100°C). The stepwise and net reactions are:
(*a*) nickel ferrite ($NiFe_2O_4$):

$$Ni^{2+} + 2Fe^{3+} + 8OH^- \rightarrow Ni(OH)_2 \cdot 2Fe(OH)_3 \rightarrow (100^oC) \rightarrow NiFe_2O_4;$$

(*b*) cobalt ferrite ($CoFe_2O_4$):

$$Co^{2+} + 2Fe^{3+} + 8OH^- \rightarrow Co(OH)_2 \cdot 2Fe(OH)_3 \rightarrow (100^oC) \rightarrow CoFe_2O_4;$$

(*c*) magnetite ($FeFe_2O_4$):

$$Fe^{2+} + 2Fe^{3+} + 8OH^- \rightarrow Fe(OH)_2 \cdot 2Fe(OH)_3 \rightarrow (100^oC) \rightarrow Fe_3O_4.$$

3. RESULTS AND DISCUSSIONS

Samples of $FeFe_2O_4$, $CoFe_2O_4$, and $NiFe_2O_4$ were analysed for their elemental composition using Energy Dispersive X-Ray (EDX) Spectroscopy. The EDX spectra of these samples are presented in Figs. 1–3.

The elemental composition of $NiFe_2O_4$ magnetic nanoparticles was determined using EDX Spectroscopy and studied in a dispersed state. The EDX spectrum obtained for $NiFe_2O_4$ magnetic nanoparticles is presented in Fig. 1. According to this figure, the sample contains Fe at 65.3% by mass, Ni at 34.5%, and Co at 0.22%.

The elemental composition of $CoFe_2O_4$ magnetic nanoparticles was determined using EDX Spectroscopy and studied in a dispersed state.

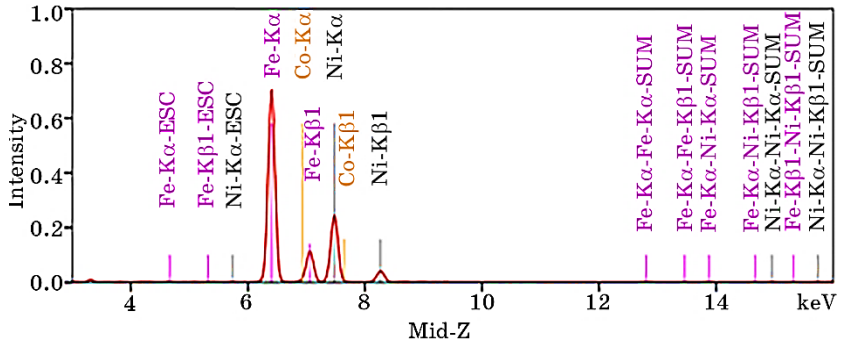

Fig. 1. EDX spectrum of powdered $NiFe_2O_4$ magnetic nanoparticles.

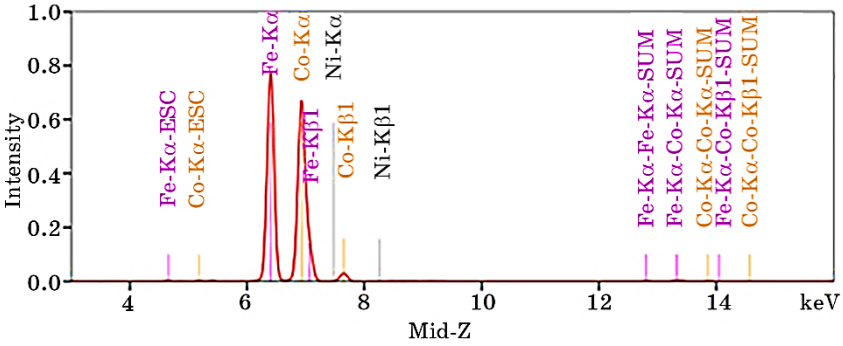

Fig. 2. EDX spectrum of powdered $CoFe_2O_4$ magnetic nanoparticles.

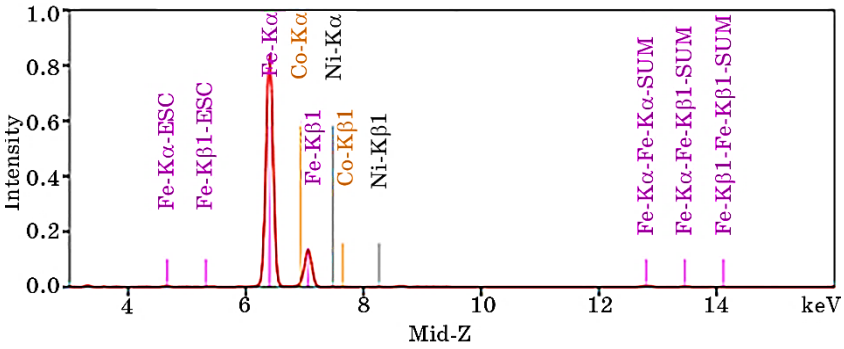

Fig. 3. EDX spectrum of powdered $FeFe_2O_4$ magnetic nanoparticles.

The EDX spectrum obtained for $CoFe_2O_4$ magnetic nanoparticles is presented in Fig. 2. According to this figure, the sample contains Fe at 61.5% by mass, Ni at 0.1%, and Co at 38.4%.

The elemental composition of $FeFe_2O_4$ magnetic nanoparticles was

determined using EDX Spectroscopy and studied in a dispersed state. The EDX spectrum obtained for $FeFe_2O_4$ magnetic nanoparticles is presented in Fig. 3. According to this figure, the sample contains Fe at 99.7% by mass, Ni at 0.1%, and Co at 0.2%. These results are presented in Tables 1–3 below.

Based on the results from Tables 1–3, it can be concluded that $NiFe_2O_4$, $CoFe_2O_4$, and $FeFe_2O_4$ magnetic nanoparticles were successfully synthesized.

The crystal structures of $NiFe_2O_4$, $CoFe_2O_4$, and $FeFe_2O_4$ magnetic nanoparticles were studied using x-ray diffraction (XRD) spectra. The $NiFe_2O_4$, $CoFe_2O_4$, and $FeFe_2O_4$ magnetic nanoparticles were dried, converted into powder form, and placed in a special container. X-ray beams with a uniform wavelength were directed onto their surfaces at angles ranging from 10° to 75° degrees. These reflected beams diffracted in the magnetic nanoparticles when positioned at an angle satisfying the Bragg condition. The dependence of the intensity of these diffracted x-ray beams on the angle of incidence on the nanoparticle surface is shown in Fig. 4 [5, 6, 9].

The structure of the magnetic nanoparticles was studied based on the relationship between the obtained intensity and the angle of incidence. As seen in Figure 4, the x-ray diffraction spectra of the synthesized magnetic nanoparticles show intensity peaks corresponding to

TABLE 1. Analysis results of the composition of $NiFe_2O_4$ magnetic nanoparticles.

No.	Element	Content	Statistical Error	Spectral Line	Intensity [cps·μA^{-1}]
1	Fe	65.3 wt.%	±0.0443%	M: FeK_α	285.91789
2	Ni	34.5 wt.%	±0.0263%	M: NiK_α	105.82723
3	Co	0.22 wt.%	±0.0061%	M: CoK_α	1.47739

TABLE 2. Analysis results of the composition of $CoFe_2O_4$ magnetic nanoparticles.

No.	Element	Content	Statistical Error	Spectral Line	Intensity [cps·μA^{-1}]
1	Fe	61.5 wt.%	±0.0372%	M: FeK_α	312.63410
2	Ni	0.1 wt.%	±0.0052%	M: NiK_α	0.40497
3	Co	38.4 wt.%	±0.0260%	M: CoK_α	279.11786

TABLE 3. Analysis results of the composition of $FeFe_2O_4$ magnetic nanoparticles.

No.	Element	Content	Statistical Error	Spectral Line	Intensity [cps·μA^{-1}]
1	Fe	99.7 wt.%	±0.0478%	M: FeK_α	425.83336
2	Ni	0.1 wt.%	±0.0038%	M: NiK_α	0.31522
3	Co	0.2 wt.%	±0.0071%	M: CoK_α	1.71781

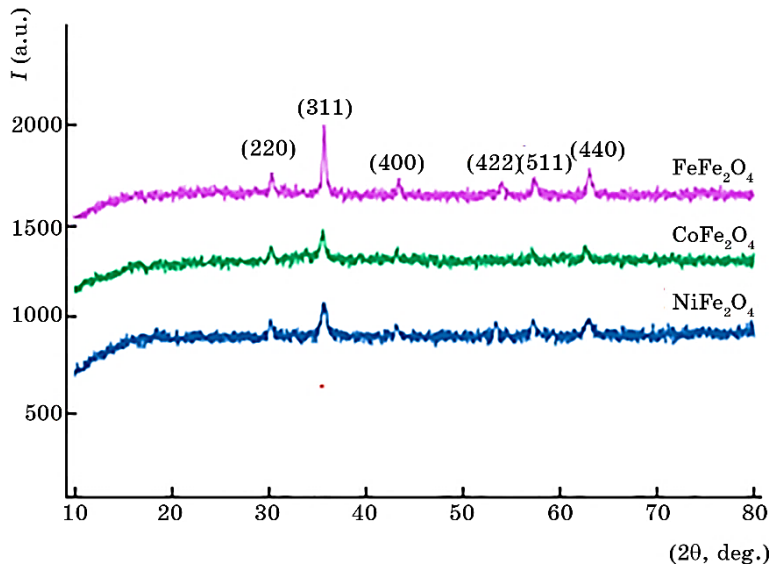

Fig. 4. X-ray diffraction of powdered $NiFe_2O_4$, $CoFe_2O_4$, and $FeFe_2O_4$ magnetic nanoparticles.

(220), (311), (400), (422), (511), and (400). The spectra of the diffracted x-rays indicate that all diffraction peaks for $NiFe_2O_4$, $CoFe_2O_4$, and $FeFe_2O_4$ magnetic nanoparticles correspond to a single-phase inverse cubic spinel structure, which is consistent with the results reported in scientific articles [3–7] (Fig. 4).

The average crystallite size of the $NiFe_2O_4$, $CoFe_2O_4$ and $FeFe_2O_4$ magnetic nanoparticles was estimated using the Debye–Scherrer equation from the most intense (311) diffraction peak in the x-ray diffraction (XRD) patterns:

$$D = \frac{0.89\lambda}{\beta\cos\theta}.$$

Here, λ is the wavelength of the x-ray radiation, β is the full width at half maximum (FWHM) of the spectral peak, and θ is the angle formed at the peak.

The lattice constants of $NiFe_2O_4$, $CoFe_2O_4$ and $FeFe_2O_4$ magnetic nanoparticles were determined using the following formula:

$$a = \sqrt{(h^2 + k^2 + l^2)d^2}\ .$$

Here, d is the distance between parallel planes, and h, k, l are the Miller's indices.

Additionally, the density of the nanoparticles was determined using

the following formula:

$$\rho = \frac{8M}{N_A a^3}.$$

Here, M represents the molecular weight of the magnetic nanoparticles, N_A is Avogadro's constant, and a is the crystal lattice parameter.

The particle size, lattice constant, and average density of the magnetic fluid particles, calculated using semi-empirical methods, are presented in Table 4 [4, 6, 9].

In this study, the composition of the magnetic fluids was formulated as follows:

1) $NiFe_2O_4$, $CoFe_2O_4$, and $FeFe_2O_4$ magnetic nanoparticles were used as the primary magnetic particles;

2) oleic acid ($C_{18}H_{34}O_2$) was employed as the surface-active agent;

3) distilled water was utilized as the liquid medium. Using these components, magnetic fluids with volumetric concentrations ranging from 0.5% to 5%, based on $NiFe_2O_4$, $CoFe_2O_4$, and $FeFe_2O_4$ magnetic nanoparticles, were prepared. Their electrical conductivity at room temperature was measured using the Karlaush method.

The magnetic fluids based on $NiFe_2O_4$, $CoFe_2O_4$, and $FeFe_2O_4$ magnetic nanoparticles investigated experimentally exhibit low electrical conductivity due to their predominantly dielectric nature. However, as the concentration of magnetic nanoparticles in these magnetic fluids increases, their electrical conductivity shows a significant rise. As observed from Fig. 5, the electrical conductivity of the prepared magnetic fluids at room temperature is as follows:

- for $FeFe_2O_4$-based magnetic fluid, with magnetic nanoparticle concentrations ranging from 0.5% to 5%, the electrical conductivity increased linearly from 15.6 μS/cm to 147.8 μS/cm;
- for $CoFe_2O_4$-based magnetic fluid, with magnetic nanoparticle concentrations ranging from 0.5% to 5%, the electrical conductivity increased linearly from 2.1 μS/cm to 20.2 μS/cm;
- for $NiFe_2O_4$-based magnetic fluid, with magnetic nanoparticle concentrations ranging from 0.5% to 5%, the electrical conductivity increased linearly from 0.23 μS/cm to 10.4 μS/cm.

The electrical conductivity of magnetic fluids is explained through

TABLE 4. Results of x-ray phase analysis of $NiFe_2O_4$, $CoFe_2O_4$, and $FeFe_2O_4$ magnetic nanoparticles.

Sample	D_{XRD} [nm]	a [Å]	a_{ave} [Å]	V [Å^3]	ρ_{XRD} [g·sm^{-3}]
$NiFe_2O_4$	16.9 ± 0.1	8.30		571.8	5.45
$CoFe_2O_4$	10.3 ± 0.1	8.37	8.47 ± 0.03	587.0	5.31
$FeFe_2O_4$	9.7 ± 0.1	8.73		598.0	5.28

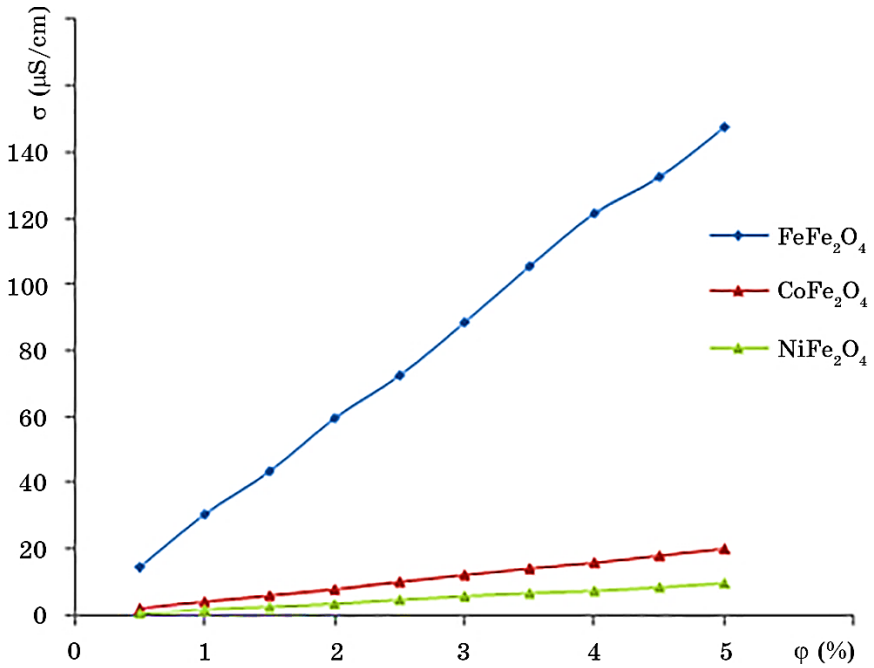

Fig. 5. Dependence of the electrical conductivity of magnetic fluids on concentration at room temperature.

an equation based on the Maxwell's model. However, for low-concentration magnetic fluids based on $NiFe_2O_4$, $CoFe_2O_4$, and $FeFe_2O_4$ magnetic nanoparticles, the enhanced Shen's model provides a more suitable explanation. According to this model, the following equation is formulated for low-concentration magnetic fluids: $\sigma = \sigma_M + \sigma_E + \sigma_B$. Here, σ_M represents the electrical conductivity calculated based on the Maxwell's model, σ_E denotes the electrical conductivity arising due to electrophoretic mobility, and σ_B indicates the electrical conductivity resulting from Brownian motion [11, 12].

4. CONCLUSIONS

$NiFe_2O_4$, $CoFe_2O_4$, and $FeFe_2O_4$ magnetic nanoparticles were synthesized *via* the chemical co-precipitation (chemical condensation) method. X-ray diffraction analysis confirmed that all diffraction peaks corresponded to a single-phase inverse cubic spinel structure for each composition, in agreement with previously reported literature data. Particle size estimation, based on transmission electron microscopy and the Debye–Scherrer equation, indicated average diameters of 25–38 nm for $NiFe_2O_4$, 10–30 nm for $CoFe_2O_4$, and 7–30 nm for $FeFe_2O_4$.

Electrical conductivity measurements at room temperature revealed the lowest value of 0.23 μS·cm^{-1} for the $NiFe_2O_4$-based magnetic fluid at 0.5% nanoparticles' concentration, and the highest value of 147.8 cm^{-1} for the $FeFe_2O_4$-based magnetic fluid at 5% concentration. These findings are consistent with predictions of the Shen's model.

AUTHORS' CONTRIBUTIONS

U. E. Nurimov carried out the experimental measurements, synthesized the magnetic nanoparticles, performed data processing, prepared the figures, and contributed to manuscript editing. O. Q. Quvondiqov conceptualized the research, supervised the study, developed the methodology, and conducted data analysis. Both authors discussed the results, contributed to the final version of the manuscript, and approved the submitted version.

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