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研究課題名(和文) Development of Multifunctional Photo-responsive Molecular Magnets

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研究成果の概要(和文)：主に磁気特性と光学特性の相関に関する研究を行った。主な成果としては、様々な磁気特性および光学特性を示す遷移金属錯体の合成と分析が挙げられる。これらの化合物は光照射下で特異な電子的・磁気的特性を示し、先端技術への応用に繋がる可能性を示唆した。さらに、配位化合物について、発光や非線形光学活性などのさまざまな物理化学的効果を調べた。また、磁性現象と光学現象の間の相関現象の探索にも努めた。これらの知見は、多機能材料の理解を進める上での光磁気研究の重要性を示すものであり、将来の技術革新の基礎を築くものである。

研究成果の学術的意義や社会的意義

本研究では特に光磁性の研究に重点を置き、物質の磁気特性と光学特性の融合を追求した。様々な化合物の合成と分析により、光照射下での特異な挙動を明らかにし、技術の進歩のみならず基礎研究にも貢献した。これらの現象の理解は、幅広い分野への応用が期待される新材料の開発に役立つと考えられ、科学の発展と社会の進歩に貢献することが期待される。

研究成果の概要(英文)：The research primarily focused on investigating the integration of magnetic and optical properties, with a particular emphasis on photomagnetic studies. Key achievements included synthesizing and analyzing transition metal complexes exhibiting various mechanisms of photomagnetic behaviors. These compounds displayed unique electronic and magnetic traits under light exposure, highlighting their potential applications in advanced technologies. Additionally, coordination compounds were examined for different physicochemical effects, such as luminescence and nonlinear optical activity. Moreover, efforts were made to explore cross-effects between magnetic and optical phenomena. These findings underscore the significance of photomagnetic studies in advancing our understanding of multifunctional materials and lay the groundwork for future technological innovations.

研究分野：Physical chemistry

キーワード：Functional materials Switchable materials Molecular magnetism Photomagnets Luminescence
Nonlinear optics Low-frequency absorption Optical materials

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1. 研究開始当初の背景

(1) The study of multifunctional molecule-based materials has emerged as a significant topic in chemistry and materials science. Specifically, molecular magnetic materials, a novel class of compounds, have garnered substantial attention over the past two decades. Unlike traditional magnets composed of metal alloys and oxides, molecular magnets are constructed from organic and inorganic elements. This unique composition renders them light, transparent, and capable of self-assembly from solution, which offers advantages for creating customizable materials.

(2) The principal investigator had a proven track record in pioneering the design, synthesis, and characterization of novel octacyanidometallate(IV)-based molecular photomagnets. These materials exhibited unique and promising magnetic properties, such as slow magnetic relaxation, spin crossover, and long-range magnetic ordering. Given this background, the motivation for the research was driven by the potential to develop new multifunctional materials that could impact fundamental research and revolutionize modern electronics, energy conversion systems, and sensors. The focus was on photo-responsive materials that combined nontrivial magnetic and photomagnetic behaviors with additional properties like nonlinear optical activity, luminescence, and ionic conductivity. The primary scientific goal was to enhance the understanding of photon-matter interactions in molecular compounds, paving the way for technological advancements and reinforcing Japan's leadership in material science research.

2. 研究の目的

The initial objective was to develop advanced multifunctional photo-responsive molecular materials. Specifically, the aim was to design and synthesize photomagnetic materials with additional functionalities such as nonlinear optical effects, ionic conductivity, and luminescence.

The research sought to integrate chiral ligands into material structures or use spontaneous resolution techniques to create enantiopure materials. These would be tested for photo-responsiveness and examined for nonlinear optical effects like circular dichroism, piezoelectricity (second-harmonic generation, SHG), and pyroelectricity (ferroelectricity), aiming to detect new cross-effects between magnetic and optical phenomena. Additionally, the research aimed to enhance ionic conductivity in photomagnetic materials by replacing heavy alkali cations with smaller cations, studying their impedance and photomagnetic properties across various conditions. Another goal was to develop luminescent photo-responsive magnets using lanthanide(III) complexes and photoreactive octacyanidometallates, exploring interactions between magnetic phenomena and luminescence parameters.

Achieving these objectives would uncover novel multifunctional photomagnetic materials with potential applications in advanced electronic and optoelectronic devices, significantly contributing to material science and technology.

3. 研究の方法

The research project employed a comprehensive approach, integrating advanced synthesis techniques, extensive characterization methods, and innovative measurement setups to achieve its objectives. The synthesis was based on a self-assembly approach using prefunctionalized complexes, which are building blocks that already possess intrinsic properties such as chirality, conductivity, luminescence, or magnetism. Basic characterization of these materials was conducted using elemental analysis, X-ray diffraction, and UV-visible and IR spectroscopies. Further development involved introducing additional structural elements or applying external physicochemical stimuli, such as desolvation. Magnetic and photomagnetic studies of polycrystalline samples were performed using Quantum Design magnetic property measurement system (MPMS) superconducting quantum interference device (SQUID) magnetometers. These magnetometers, equipped with optical sample rods and various laser sources spanning the visible and near-IR spectrum, allowed for direct illumination of the samples to detect light-matter interactions. To investigate nonlinear optical effects, chiral magnetic materials were synthesized by incorporating chiral ligands (e.g., alkaloids) or using achiral components with a predisposition for spontaneous resolution (e.g., *N,N*-dimethylacetamide, triphenylphosphine oxide). These materials were studied using

circular dichroism spectroscopy and second harmonic generation detection setups combined with liquid helium-cooled cryostats. For ionic conductivity studies, lithium salts of octacyanidometallates were synthesized and reacted with copper(II) complexes to produce extended systems with ionic conductance. These materials were analyzed using impedance spectroscopy at different temperatures and humidity levels to study their electrical performance before and after irradiation. Luminescent systems were prepared using lanthanide(III) complexes with intrinsic luminescence properties. These materials were thoroughly examined using spectrofluorometers to detect color, intensity, lifetime, and quantum yield. Additionally, the low-frequency absorption properties of the materials were investigated using THz-TDS (terahertz time-domain spectroscopy) spectrometers and low-frequency Raman spectroscopy. This holistic approach ensured a thorough exploration and characterization of the new multifunctional materials, aiming to uncover and document novel physicochemical phenomena.

4. 研究成果

The research project focused on developing and characterizing multifunctional photomagnetic materials, achieving significant advancements in both magnetic and other physicochemical properties.

(1) The primary accomplishments of this project include the synthesis and detailed analysis of various transition metal complexes with multifunctional properties. Key highlights involve the development of desolvation-assisted spin-crossover (DASCO) and light-induced excited spin-state trapping (LIESST) in Fe(II) complexes [*Inorg. Chem. Front.* 8, 3210 (2021)]. Two new Fe(II) complexes, $[\text{Fe}(\text{3-bpp})_2](\text{ClO}_4)_2 \cdot 2.5\text{H}_2\text{O} \cdot \text{MeOH}$ and $[\text{Fe}(\text{3-bpp})_2](\text{ReO}_4)_2 \cdot 3\text{H}_2\text{O}$, 3-bpp = 2,6-di-(1*H*-pyrazol-3-yl)pyridine, demonstrated SCO from diamagnetic low-spin ($S = 0$) to paramagnetic high-spin ($S = 2$) states at around 340 K, with reversible spin conversion at lower temperatures of 210 K and 304 K, respectively. Photomagnetic studies for the first compound confirmed the LIESST phenomenon with relaxation temperatures $T_{\text{LIESST}} = 82$ K and 66 K for the solvated and desolvated phases, respectively. For the second material, no LIESST effect was observed for the solvated sample, but a LIESST behavior at $T_{\text{LIESST}} \approx 50$ K was detected for the desolvated compound. This highlights their potential applications in temperature-responsive materials and solvent sensors. Additionally, research on similar complexes showing spontaneous resolution is underway.

(2) Next, the research investigated the synthesis and properties of coordination compounds involving octacyanidomolybdate(IV) ions and copper(II) complexes with chelating ligands [*Inorg. Chem. Front.* 9, 771 (2022)]. Two newly synthesized compounds $\{[\text{Cu}(\text{tren})_2(\mu\text{-tn})] \cdot [\text{Mo}(\text{CN})_8] \cdot 7.5\text{H}_2\text{O}$ and $\{[\text{Cu}(\text{tren})_2(\mu\text{-tn})] \cdot [\text{Mo}(\text{CN})_8] \cdot \{[\text{Cu}(\text{tren})_2[\text{Mo}(\text{CN})_8]\} \cdot 9\text{H}_2\text{O}$, tren = tris(2 aminoethyl)amine and tn = 1,3-diaminopropane, along with two reference systems $[\text{Cu}(\text{tn})_2]_2[\text{Mo}(\text{CN})_8] \cdot 2\text{H}_2\text{O}$ and $[\text{Cu}(\text{tren})_2]_2[\text{Mo}(\text{CN})_8] \cdot 5.25\text{H}_2\text{O}$ were analyzed. Spectroscopic and quantum chemical analysis revealed distinct electronic transitions and magnetic studies indicated paramagnetic behavior with weak antiferromagnetic interactions. Photomagnetic effects were observed for irradiation with 473 and 410 nm lights at 10 K, with the first compound showing a LIESST effect on Mo(IV) centers from diamagnetic low-spin ($S = 0$) to paramagnetic high-spin ($S = 1$) states, while others displayed metal-to-metal charge transfer (MMCT) in Cu(II)-Mo(IV) pairs. This research sheds light on the complex behaviors of these coordination compounds, integrating coordination chemistry, spectroscopy, quantum chemistry, and magnetic studies. Unexpectedly, three-dimensional networks $\{[\text{Cu}_3(\mu_3\text{-OH})(\mu\text{-pz})_3(\text{H}_2\text{O})_3]_2[\text{M}(\text{CN})_8]\} \cdot n\text{H}_2\text{O}$ ($M = \text{W}$ and Mo), Hpz = pyrazole, composed of trinuclear triangle copper(II) (TTC) secondary building unit and octacyanidometallates(IV) [*Inorg. Chem.* 61, 8930 (2022)], showed strong antiferromagnetic interactions within the TTC units at ambient temperature, spin frustration with antisymmetric magnetic exchange-coupling constants of about +32 and +46 cm^{-1} at low temperatures, and no photomagnetic responses, indicating that the interaction between light and matter in the Cu(II)-Mo(IV) systems needs further investigation.

(3) Further studies explored spin-crossover (SCO) and charge-transfer (CT) phenomena, crucial for developing materials with advanced memory and sensing capabilities. While most SCO systems rely on iron(II) complexes, cobalt(II)-2,2':6,2''-terpyridine (Co-terpy) systems offer promise as alternatives. The research synthesized and characterized two new complex salts: $[\text{Co}^{\text{II}}(\text{terpy})_2]_2[\text{Mo}^{\text{IV}}(\text{CN})_8] \cdot 15\text{H}_2\text{O}$ and $[\text{Co}^{\text{II}}(\text{terpy})_2]_3[\text{W}^{\text{VI}}(\text{CN})_8] \cdot 12\text{H}_2\text{O}$. Structural analysis of both compounds revealed layers of $[\text{Co}(\text{terpy})_2]^{2+}$ pillared by octacyanidometallate anions and interspersed with water

molecules [*Eur. J. Inorg. Chem.* 26, e202300307 (2023)]. Magnetic studies confirmed partial SCO on the cobalt(II) centers from paramagnetic low-spin ($S = 1/2$) to paramagnetic high-spin ($S = 3/2$) states in both hydrated and desolvated phases, with humidity-induced SCO observed for the Co(II)-Mo(IV) system and a new phenomenon termed isothermal humidity-activated charge-transfer-induced spin transition (HACTIST) for the Co(II)-W(V) system: $\text{Co}^{\text{II-HS}}(S = 3/2)\text{-W}^{\text{V}}(S = 1/2)$ $\text{Co}^{\text{II-LS}}(S = 0)\text{-W}^{\text{IV}}(S = 0)$. These findings elucidate the water-solvation-dependent spin transitions in Co(II)-octacyanidometallate(IV/V) complexes. Although the photomagnetic LIESST effect is not observed on cobalt(II) centers, preliminary tests indicate LIESST on Mo(IV) centers and photoinduced charge transfer in Co(III)-W(IV) pairs at low temperatures.

(4) In the area of linear and nonlinear optical properties, one-dimensional zigzag cyanide-bridged coordination polymers $[\text{Ln}^{\text{III}}(\text{dma})_5][\text{W}^{\text{V}}(\text{CN})_8]$ ($\text{Ln} = \text{Gd-Lu, Y}$), dma = *N,N*-dimethylacetamide, were prepared [*Inorg. Chem.* 60, 12009 (2021)]. They crystallized in the noncentrosymmetric space group $P2_1$. Magnetic studies revealed weak antiferromagnetic interactions *via* CN⁻bridges, forming ferrimagnetically coupled chains at very low temperatures. *Ab initio* calculations provided crystal field parameters for Ln(III) ions, enabling the determination of magnetic coupling constants ranging from -1 to -5 cm^{-1} by fitting temperature-dependent magnetic susceptibilities. These compounds exhibited wide optical transparency and demonstrated second harmonic (SH) generation properties with SH susceptibilities ranging from 4.7×10^{-12} to 9.4×10^{-11} esu. Comparison with theoretical calculations and previous reports suggested a potential relationship between the lanthanide ion type and SH intensity. Additionally, these materials are investigated for lanthanide-centered luminescence and photomagnetic effects.

(5) Another study focuses on the development of multifunctional oriented polar crystals that integrate optical, nonlinear optical, and magnetic properties. Two enantiomorphic assemblies, $[\text{Yb}^{\text{III}}(\text{TPPO})_3(\text{NCE})_3]$ ($E = \text{S or Se}$), TPPO = triphenylphosphine oxide, were synthesized [*Adv. Opt. Mater.* 10, 2101721 (2022)]. Both crystallites exhibited low-frequency (LF) Raman scattering at 16 cm^{-1} (0.48 THz), arising from collective vibrations of the TPPO ligand and pseudohalides, as supported by *ab initio* calculations. Additionally, the crystals demonstrated high-intensity second harmonic generation (SHG) along the threefold rotation *c*-axis. Polarization-variation experiments on single crystals offered insight into the tensor elements constituting the $R3$ space group. Substituting thiocyanide with SeCN enhanced the second harmonic signal due to increased polarization. Furthermore, these crystals exhibited high-performance near-infrared luminescence thermometric properties originating from 4f-metal centers and weak single-molecule magnet behaviors. They serve as a platform for studying magnetic and linear/nonlinear optical cross-effects.

(6) Moreover, the research explores isomers formed by the self-assembly of iron(II) and tetra(thiocyanato)mercury(II) ions in excess 2-acetylpyridine (2-acpy) [*Inorg. Chem.* 62, 3278 (2023)]. Two isomers with the same chemical formula $[\text{Fe}(2\text{-acpy})][\text{Hg}(\mu\text{-SCN})_4]$ were identified: one with two-dimensional centrosymmetric layers and another with three-dimensional chiral networks with right- or left-handed helices. Novel synthesis methods for functional thiocyanate-bridged materials were proposed. Comparison of isomers revealed subtle spectroscopic differences. The second isomer demonstrated spontaneous resolution and exhibited nonlinear optical activity, confirmed through circular dichroism and second harmonic generation studies. Magnetic analysis showed high-spin iron(II) behavior at low temperatures, providing insights into magnetic anisotropy vectors. *Ab initio* calculations supported the observed structural and optical properties.

(7) The principal investigator (PI) endeavored to synthesize the lithium analogue of the photomagnetic compound $\text{K}_4\{[\text{Cu}(\text{ida})]_2[\text{Mo}(\text{CN})_8]\} \cdot 4\text{H}_2\text{O}$. Impedance studies indicated that the potassium compound is an insulator. Despite preparing the lithium salt precursor of $[\text{Mo}(\text{CN})_8]$, challenges arose in obtaining an isostructural material containing lithium ions. Further investigations are required to explore alternative synthetic pathways.

Domestically, this research stands among the leading work in the field of multifunctional photomagnetic materials. The innovative methodologies and novel materials developed have been recognized through high-impact publications and presentations at major conferences. Internationally, the project's contributions have been acknowledged at several conferences, with significant potential applications as well as implications for fundamental research.

The future of this research holds promise, with several avenues for further exploration.

Continued examination of Fe(II)-Hg(II) networks is predicted to yield novel functional materials. Concurrent investigations into Cu(II) polynuclear clusters, combined with photoreactive $[\text{Mo}(\text{CN})_8]^{4-}$ anions, aim to uncover new photomagnetic materials with enhanced properties. Additionally, further exploration of lanthanide(III) complexes will concentrate on elucidating their luminescent and thermometric properties, which hold practical applications in bioimaging and other fields.

Unexpected occurrences during the project have led to valuable insights. The discovery of spin-crossover properties in Fe(II)-Hg(II) networks has opened new avenues for research into temperature- and photo-responsive materials. Furthermore, the mechanical exfoliation of similar systems into nanosheets has underscored the potential for developing nanomaterials with unique mechanical and optical characteristics. Overcoming challenges has also spurred the development of new experimental methodologies for studying phenomena such as low-frequency THz absorption and Raman scattering, deepening our understanding of the photomagnetic functionality of these materials.

In conclusion, this research project has achieved significant milestones in synthesizing and characterizing multifunctional photomagnetic materials. It has propelled the understanding of these materials forward and established a foundation for future innovations in memory, sensing, and other high-tech applications. The project's findings have had substantial impacts both domestically and internationally, offering a promising outlook for the future and invaluable insights gathered from discoveries.

5. 主な発表論文等

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1. 著者名 Jedrzejowska Katarzyna, Kobylarczyk Jędrzej, Tabor Dominika, Srebro-Hooper Monika, Kumar Kunal, Li Guanping, Stefanczyk Olaf, Muziol Tadeusz M., Dziedzic-Kocurek Katarzyna, Ohkoshi Shin-ichi, Podgajny Robert	4. 巻 63
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〔図書〕 計0件

〔産業財産権〕

〔その他〕

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6. 研究組織

氏名 (ローマ字氏名) (研究者番号)	所属研究機関・部局・職 (機関番号)	備考
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7. 科研費を使用して開催した国際研究集会

〔国際研究集会〕 計0件

8 . 本研究に関連して実施した国際共同研究の実施状況

共同研究相手国	相手方研究機関
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