



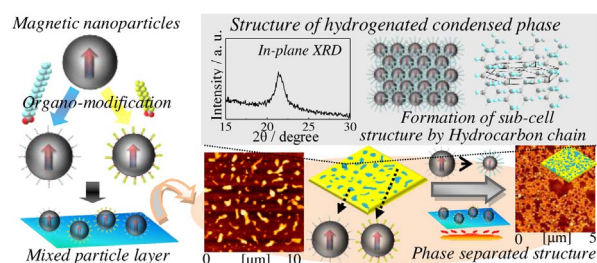
# Controlling the phase-separated morphology of a two-dimensional integrated layer of magnetic nanoparticles by surface modifications using immiscible amphiphiles



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## GRAPHICAL ABSTRACT



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## ABSTRACT

Surface modification with immiscible surfactants was utilized to induce phase separation at the nanometer scale in a two-dimensional particle layer of magnetic nanoparticles. Cobalt ferrite ( $\text{CoFe}_2\text{O}_4$ ) particles (diameter = 30 nm) and magnetite ( $\text{Fe}_3\text{O}_4$ ) particles (diameters = 5 and 30 nm) were typically subjected to surface modification with a hydrogenated and fluorinated long-chain carboxylic acid. A mixed monolayer of hydrogenated and fluorinated organo-magnetic nanoparticles was spread at the air/water interface. This system was used to assess the phase separation because the collapsed surface pressures of both components were individually observed in the isotherms that were measured by systematically changing the composition ratio. A nano-sized phase separation morphology was observed on the surface of the mixed single-particle layers by atomic force microscopy. A “sea-island” structure was observed in which the expanded phase formed by the fluorinated organo-magnetic nanoparticles surrounded the condensed nano-domains of the hydrogenated organo-magnetic nanoparticles. The separate (particulate) nano-phase morphology showed a temperature dependence, and in this case, the fluorinated “sea” phase transformed into a network morphology. The nano-domain of the hydrogenated organo-modified magnetic nanoparticles was a crystalline phase in which the modified chain was packed with two-dimensional hexagonal or orthorhombic systems. The nanophase separation on the surface of the magnetic single-nanoparticle layers likely formed because of repulsive interactions between the immiscible surface modifiers.

**Abbreviations:** AFM, atomic force microscopy; FT, IR Fourier-transform infrared; LB, Langmuir-Blodgett; MNP, magnetic nanoparticle; NP, nanoparticles; oMNP, organo-modified magnetic nanoparticles; TG, thermogravimetry; XRD, X-ray diffraction

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## 1. Introduction

A number of qualities and processes (e.g., miscibility [1], immiscibility [2], phase separation [3], and adsorption [4]) are manifested at the contact interface of composite [5] and hybrid [6] materials, and in mixed molecular systems [7]. These can open up new applications for devices, for example, in the areas of nanolithography [8] and multipatterning [9]. Studies of the phase separation of inorganic fine particles [10] have been performed to create new functional materials by combining various physical properties (e.g., particle diameter [11] and optical properties [12]) of the fine particles. In particular, the ratio of the diameter of the fine particles used in the structural formation is quite important [13]. Materials with magnetic properties (one type of inorganic fine particles [14]), discrete track media [15] (for the reduction of memory noise by phase separation and embedding a non-magnetic layer of several tens of nanometers into the magnetic layer), etc., are expected to have applications for magnetic storage media [16]. However, it is difficult to control the fine structure of materials with such magnetic properties because of the remarkable cohesion from the strong Coulomb interactions [17] among the particles.

The phase separation of organic molecules typified by general purpose polymer materials [18], e.g., phase separation in polymer materials, typically forms a structure such as a sphere [19], rod [20], gyroid [21], or lamella [22]. Moreover, hydrocarbon and fluorocarbon chains are generally immiscible when they are included in the same system [23]. When a phase-separated structure forms in such systems, the expanded fluorocarbon chains form a “sea” around the aggregated hydrocarbon “islands.”<sup>24</sup> Many widely cited and important studies have used its immiscibility for phase separation in any organic materials including polymers [25,26]. As described above, the phase separation formed by the organic molecules can be diversified and more easily controlled than that formed by inorganic materials.

Therefore, we used organo-modifications on the outermost surface of inorganic fine particles [27–31] in this study. It is known that van der Waals interactions are weaker for long alkyl chain groups bound to inorganic fine particles than between organic molecules. Moreover, fabrication of integrated layers of inorganic fine particles such as clay [27], zirconia [28], zinc oxide [29], and nanodiamond [30] have been attained by utilizing the affinity between hydrophobic chains. In particular, within a system of organized magnetic nanoparticles (MNPs), the formation of interfacial particle layers was achieved with very few

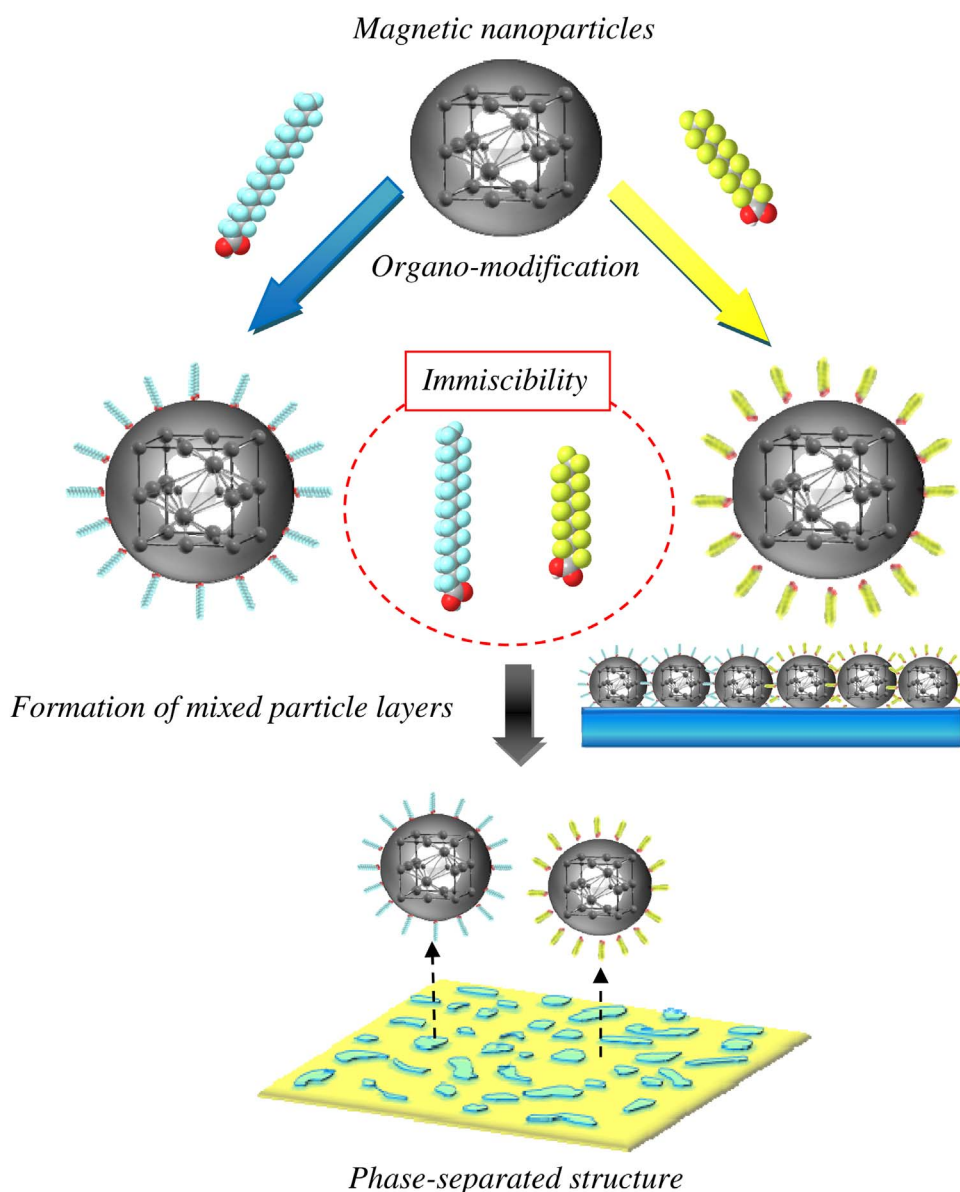


Fig. 1. Schematic illustration of the formation of a mixed particle layer by organo-modified magnetic nanoparticles.

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