



Synthesis and characterization of $\text{Mn}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ and Fe_3O_4 nanoparticle ferrofluids for thermo-electric conversion

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ABSTRACT

Ferrofluids containing nanoparticles of $\text{Mn}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ (MZ5) and Fe_3O_4 (magnetite) have been examined as potential thermal transport media and energy harvesting materials. The ferrofluids were synthesized by chemical co-precipitation and characterized by EDX to determine composition and by TEM to determine particle size and agglomeration. A range of particle coatings and carrier fluids were used to complete the fluid preparation. Commercially available ferrofluids were tested in custom built rigs to demonstrate both thermal pumping (for waste heat removal applications) and power induction (for power conversion and energy harvesting applications). The results indicate that simple ferrofluids possess the necessary properties to remove waste heat, either into thermal storage or for conversion to electrical power.

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1. Introduction

A ferrofluid is a colloidal suspension of ferromagnetic nanoparticles held within a carrier fluid [1]. The carrier can be a solvent, hydrocarbon oil or aqueous liquid dependent on use. Long chain surfactants, for example oleic acid, are used to coat the nanoparticles in order to inhibit flocculation [2]. As the nanoparticles are around 10 nm in size they are subject to Brownian motion which keeps them suspended in the carrier fluid. Their uses include the role of a cooling medium in loudspeakers and high power transformers, inkjet printer fluid, hard drive motor bearings, mineral separation, and (since they have a high reflectivity) as an adjustable surface fluid mirror [3–7]. The combination of suspended magnetic particles and carrier fluid give the resulting colloid the ability to change fluidic properties, most notably viscosity, in the presence of an external magnetic field. The carrier fluid, a dominant part of the system, has a great impact on physical properties such as viscosity and evaporation [8]. Since the ferromagnetic particles are of nanometer dimension, the long range ordering produces only single domains and fixed dipole magnetic fields [9]. The suspended nanoparticles in a ferrofluid, having magnetic single-domains, possess a magnetic moment, μ . When subjected to an applied external magnetic field, B , the magnetic moments have a potential energy, U , given by Eq. (1). In order to reduce U the nanoparticles orient parallel to the applied magnetic field lines. When removed from the field the

magnetic moments rapidly become randomised and magnetization becomes zero [10]

$$U = -\mu B \quad (1)$$

If the applied magnetic field has a gradient then the nanoparticles will experience a force, F , and move towards the region of greatest field intensity. This force is proportional to the applied magnetic field, B , and the magnetic moment of the nanoparticle, μ :

$$F = \nabla B \mu \quad (2)$$

Another important parameter that affects the magnetic properties of ferrofluids is the Curie temperature, T_c . Above T_c the atomic order within the magnetic domain is disrupted by thermal vibrations; the thermal energy overcomes the electronic exchange forces, reducing the saturation magnetization of the nanoparticles to zero as the disorder within the domains increases.

Heat transfer and pumping in ferrofluids has been extensively reported for both free convection and forced convection conditions. In the case of thermomagnetic free convection, experimental investigations with a varying magnetic field were published by Kikura et al. [11] and Sawada et al. [12], although the orientation of the magnetic field is unclear. Theoretical studies (analytical and numerical) of thermomagnetic free convection often suffer from shortcomings in the modeling of the magnetic field. For example, Finlayson [13], Lange [14] and Krakov and Nikiforov [15] all assumed a spatially uniform magnetic field, which is rarely the case with commercial dipoles. Similarly imprecise assumptions on the magnetic vector limit the applicability of the outputs from the theoretical work of Yamaguchi et al. [16] and Tangthieng et al. [17]. More fundamental issues are evident in the theoretical

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simulations of thermomagnetic forced convection, most notably in the analysis of Aihara et al. [18] and Yamaguchi et al. [19], where the magnetic fields are in conflict with Maxwell's equations. This was corrected by Ganguly et al. [20] who showed that the addition of line-source dipoles enhances heat flow, to a degree dependent on dipole strength and the geometry of the magnet (or the positioning of an electromagnet). Lajvardi et al. [21] designed an experiment to study the forced thermomagnetic convection and heat transfer performance of magnetite (Fe_3O_4) nanoparticles in distilled water, attributing the measured increase in the local heat transfer coefficient in the presence of a magnetic field to either an increased thermal conductivity or specific heat capacity. Self-powered thermomagnetic convection loops (self-regulated pumping) using Mn–Zn based ferrofluids have been reported by Xuan and Lian [22], but did not include any measurement of the pump pressure achieved, except to state that it must exceed the viscous drag. Ferrofluid pumping in both time-varying and spatially-travelling magnetic fields has been modeled numerically by Zahn and Greer [23] and Mao and Koser [24], with the latter calculating ferrofluid flow velocities on the order of 5–10 mm/s. Some values of pressure differences in experimental ferrofluid pumps have also been published, together with limiting effects. Later work by Mao and Koser [25] reports 1300 Pa in 2 mm $\times$ 0.25 mm microchannels. This compares with 177 Pa in 1 mm $\times$ 0.4 mm microchannels from Xuan and Lian [26], when the fluid was deteriorating owing to bubble formation. Short term operation (limited by thermal instabilities) produced a maximum pressure of 345 Pa in 2 mm diameter glass tubing in the magnetite ferrofluid investigated by Pal et al. [27].

In this work we describe the synthesis of $\text{Mn}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ (MZ5) and Fe_3O_4 (magnetite) nanoparticles by chemical co-precipitation, their subsequent coating with oleic acid, and their suspension in ethylene glycol and heptane.

Various material properties of comparable commercial ferrofluids are derived, before the suitability of the fluids for energy harvesting applications is investigated.

2. Materials and methods

To synthesize the magnetite nanoparticles the following process was followed; two 0.5 M aqueous solutions one of ferric chloride hexahydrate (98%, Aldrich) and the other ferrous chloride tetrahydrate (99%, Aldrich) were prepared and mixed together at room temperature. The resulting solution was added rapidly to a 4 M NH_4OH (30%, Sigma-Aldrich) solution heated to 80 °C under constant stirring using a non-magnetic agitator. The resulting black precipitate mixture was left to stir for 1 h at 80 $\pm$ 10 °C.

To synthesize the MZ5 nanoparticles, 0.1 M aqueous solutions of manganese chloride (98%, Aldrich), zinc sulfate heptahydrate (99%, Aldrich) and a 2 M solution of ferrous chloride tetrahydrate (99%, Aldrich) were prepared and mixed together at room temperature. The mixture was added rapidly to a 3 M solution of NaOH (97%, Aldrich) under constant stirring with a non-magnetic agitator. The resulting mixture was left to stir for 1 h at 80 °C.

Fig. 1 shows a micrograph of oleic acid coated magnetite particles, with typical size in the 10–18 nm range. The capping agent for both solutions was prepared by mixing oleic acid (90%, Sigma Aldrich) with NH_4OH in a 1:10 ratio by volume. A small quantity of this mixture was added to the magnetite and MZ5 mixtures and left to stir for a further 30 min at 80 °C.

The resulting precipitates were separated from solution by placing a magnet below the reaction vessel and pouring out the waste fluid. The magnet was then removed and the precipitate washed with deionized water, the waste being removed as above, the process being repeated multiple times. The final products

were then dispersed in hydrazine for the magnetite particles and ethylene glycol for the MZ5 particles. Fig. 2 is an EDX plot of composition for coated magnetite nanoparticles.

The concentration and type of the suspended nanoparticles, the surfactant, and the carrier fluid combine to impart the overall magnetic and rheological properties of the ferrofluid, most notably the magnetization and base viscosity. This offers the opportunity to tailor the ferrofluid to act in a closed system to provide the removal of thermal energy and its transport into a thermo-electric converter. This can be achieved in a two-stage process, as described below.

The first stage comprises a thermal pump, shown conceptually in Fig. 3.

Ferrofluid is drawn into the heat affected zone where its temperature is raised, whereupon the magnetic properties of the fluid diminish. The creation of the demagnetized fluid, coupled with the cooler highly magnetized fluid behind the hot zone, creates a pressure differential given by [28]

$$\Delta P = \mu_0 H [M(T_{\text{out}}) - M(T_{\text{in}})] \quad (3)$$

where μ_0 is the free space permeability, H is the externally applied magnetic field intensity, and $M(T)$ is the temperature

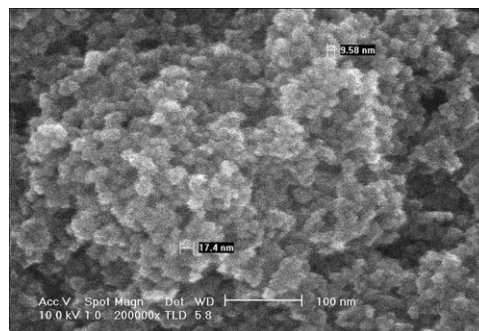


Fig. 1. TEM of coated magnetite nanoparticles.

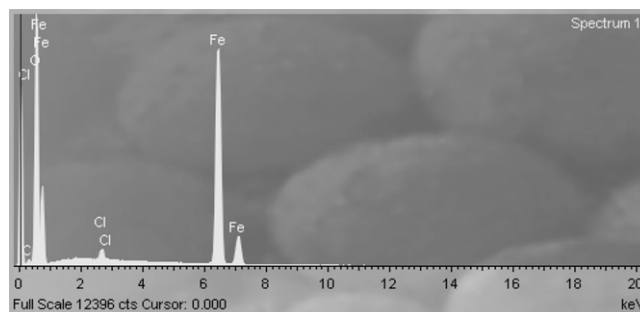


Fig. 2. EDX analysis of coated magnetite nanoparticles.

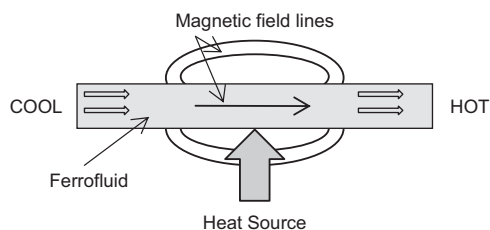


Fig. 3. Ferrofluid as a waste heat collector, creating a pressure differential in the presence of an external magnetic field.

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