## **CHAPTER 1**

## PROBLEM STATEMENT AND INTRODUCTION

A ferromagnetic liquid or ferrofluid refers to a stable colloidal dispersion or suspension of single domain magnetic particles in a carrier liquid. The suspension of particles is coated by a surfactant and suspended in a carrier liquid. The ferrofluid as referred to in this dissertation is produced in three main stages: oxide precipitation and magnetite formation, saponification of the surfactant and peptization of the coated particles. Figure 1.1 gives the schematic representation of a potential method for manufacturing ferrofluid. Magnetite (Fe<sub>3</sub>O<sub>4</sub>), a ferrimagnetic material, is produced in a precipitation reaction in reactor 3 from ammonium hydroxide (from storage tank 1) and iron salt (from storage tank 2) solutions. The magnetite is coated with oleic acid (from storage tank 4) as the surfactant and is suspended in kerosene (from storage tank 5) as the carrier liquid in the peptization stage. This occurs in reactor 6. Storage tank 7 is a vessel for housing the final product.

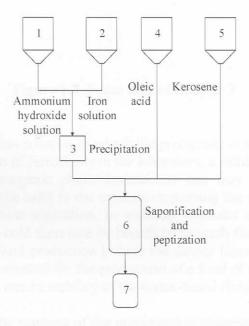


Figure 1.1 Schematic representation of a potential method for manufacturing ferrofluid

From this brief description, it appears that the process for the synthesis of ferrofluids is quite simple. However, some of the basic aspects and key manufacturing parameters are not understood fully. This dissertation attempts to explain and offer a more detailed understanding of some of these aspects.

Magnetite formation is vital for the production of ferrofluids. The formation and transformation pathways of iron oxides required to form magnetite are, however, complex. There are many

pathways that could lead to the formation of incorrect species. In Chapter 3, possible mechanisms for the formation of magnetite are discussed. Parameters that could affect the production of magnetite are discussed and an investigation is then conducted to confirm what the preferred conditions are for the production of magnetite and whether or not these parameters are of importance in ferrofluid production. Chapter 3 focuses on the section as highlighted in Figure 1.2.

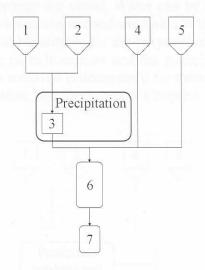


Figure 1.2 Focus area of Chapter 3

After precipitation, the aqueous solution in which the precipitate is suspended contains dissolved salts. Often, in the production of ferrofluids in the laboratory, a third phase has been produced. It is suspected that organic-inorganic phase interactions that may cause this third phase are promoted in some cases by the salts in the mixture containing the precipitate. This may hinder the final organic/aqueous phase separation. In addition, the salts in solution may increase the viscosity of the product. It would therefore be beneficial to wash the precipitate to remove these salts. For water-based ferrofluid production (where the carrier liquid is water), concentration of the magnetite suspension is essential for the production of a fluid of suitable magnetic properties. Washing is also important to ensure stability of the water-based fluids.

In Chapter 4, a method for the washing of the precipitate is suggested, making use of what have been termed sedimentation funnels. Precipitate is fed to four cone-shaped vessels in series. The funnels are plastic containers around which copper coils are wound. A direct current is passed through the coils thus generating a magnetic field in the interior of the funnel. When placed in the funnel and exposed to the magnetic field, the magnetite is attracted towards the region of greatest magnetic field and becomes concentrated at the base of the funnel. Wastewater leaves the funnels via the overflow. Computational fluid dynamics (CFD) is used to investigate the suitability of the funnel design and to determine whether the funnel configuration would ensure that the salts would be washed from the precipitate and that the magnetite would be retained in the funnels.

Chapter 5 investigates an alternative to the method of the sedimentation funnels to improve the washing and concentration of the magnetite precipitate. This concept is entitled the wet high intensity magnetic separator (WHIMS). The procedure used for the WHIMS is analogous to that of deep bed filtration with the magnetic force assisting in particle capture. The WHIMS consists of a funnel shaped vessel containing a magnetisable matrix or mesh and surrounded by a magnet e.g. solenoid electromagnet. Magnetic particles (such as the magnetite) fed to the WHIMS are captured in the matrix when the current to the coils is switched on. The liquid in which the magnetite is contained passes through the vessel. Water can be added to the vessel to wash the magnetite. In this chapter, an investigation is conducted on a WHIMS prototype that was built to determine whether or not it would be suitable for the purposes of washing and concentrating the magnetite. The efficiency of the particle capture and the particle retention on the magnetisable matrix once the magnetic field is removed is determined for three types of mesh in order to select the most suitable mesh for operation. The focus area of Chapters 4 and 5 is shown in Figure 1.3.

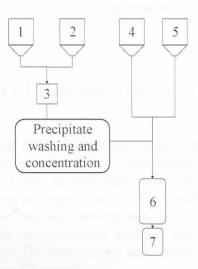


Figure 1.3 Focus area of Chapters 4 and 5

The particle size of the magnetite which is one of the most important components of the ferrofluid is critical for fluid stability. Particles must be small enough not to settle or agglomerate as a result of gravitational and magnetic interaction forces. In addition to ensuring that the particle size is correct, the coating of the particles is an important factor for maintaining stability. The coating of particles prevents agglomeration that could occur as a result of Van der Waals interaction forces. In Chapter 6, the stability requirements in terms of coating of particles are discussed briefly. An investigation is conducted to obtain an indication of the optimum quantity of surfactant required for steric stabilisation by varying the percentage surfactant added.

Chapter 7 investigates the use of a packed column for the saponification and peptization stages of the ferrofluid production. In order to achieve a more rapid phase transfer, heat is required to be input into the system. To obtain the maximum heat transfer at the maximum allowable temperature, the surface area in the column should be at a maximum. This chapter documents the procedure of mathematical optimisation using the dynamic trajectory method to determine the

optimum dimensions of such a column that would result in maximum heat transfer. The focus area of Chapters 6 and 7 is shown in Figure 1.4.

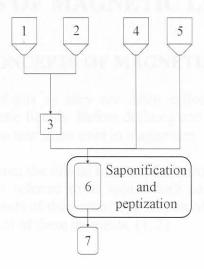


Figure 1.4 Focus area of Chapters 6 and 7

In this dissertation, each chapter contains sections discussing the theoretical background, experimental investigation, results, discussion, conclusions and recommendations. The dissertation was structured in this way to maintain clarity through the chapters. The subject matter from chapter to chapter varies significantly and it was decided to rather separate and discuss concepts individually than use the conventional format usually applied to dissertations i.e. a single introductory chapter, experimental section, results and discussion section and conclusions and recommendations. The conclusions and recommendations from all the chapters are summarised in the final chapter, Chapter 8.

A continuous plant has been designed for the production of ferrofluid on a larger scale. Some of the values used in the chapters relate to the continuous plant design and are not arbitrarily chosen. In many cases the origin of these values is not specified as the mass balance is considered to be confidential information