CHAPTER I
INTRODUCTION

1.1 Background

In the modern era of science and technology, nanomaterials have emerged as a highly intriguing and significant research subject. Nanomaterials are materials whose characteristic length scale falls between 1 and 100 nm, and whose nanoscale dimensions cause at least one attribute to differ significantly from its bulk equivalent (Donegá, 2014). At the nanometer scale, materials differ from their bulk counterparts in terms of their physical, chemical, and biological properties. Those developed at the nanoscale have more performance sensitivity than those developed at the bulk level. This is mostly because nanomaterials have a higher specific surface area, which raises their levels of reactivity (Jeevanandam et al., 2018).

Based on their size, origin, structural configuration, pore size, and possible toxicity, nanomaterials can be categorized into five groups. Four major kinds of nanomaterials can be distinguished based on their structural configuration: organic, inorganic, carbon-based, and composite. Inorganic nanomaterials are typically classified as those composed of metal-based or metal oxide-based nanomaterials. Metal oxide nanomaterials are composed of negative oxygen ions and positive metallic ions (Mekuye & Abera, 2023). Compared to other metal oxide nanomaterials, iron oxides have high adsorption capacity, surface reactivity, magnetic properties, and catalytic efficiency due to biocompatibility, superparamagnetism, and ultrafine size (Revathy et al., 2023).

In nature, iron oxides come in three main forms: magnetite (Fe_3O_4), maghemite (γ-Fe_2O_3), and hematite (α-Fe_2O_3) (Wu et al., 2015). Magnetite (Fe_3O_4) is one of the minerals in the iron oxide group renowned for having a cubic crystal structure and exceptionally strong magnetic characteristics. Regarding its structure, Fe_3O_4 nanomaterials exhibit an inverse spinel cubic crystal arrangement, as depicted in Figure 1. The molecular formula for Fe_3O_4 can be represented as (Fe^{2+}) (Fe^{3+})2O_4. From Figure 1, it can be observed that there are 8 divalent Fe^{2+} cations occupying octahedral positions, 8 trivalent Fe^{3+} cations occupying tetrahedral positions, and another 8 Fe^{3+} cations residing in octahedral positions. Despite having opposite orientation, the Fe^{3+} cations at the tetrahedral and octahedral sites possess equal and significant resultant magnetic moments(Khan et al., 2019).

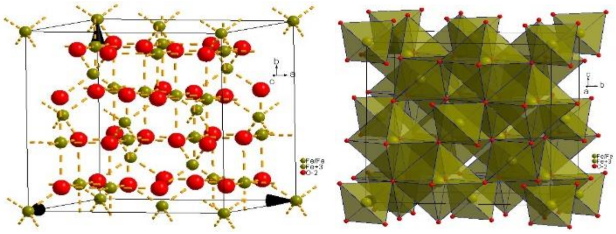

Figure 1. Structure of Fe_3O_4 nanomaterials (Khan et al., 2019)

Magnetite nanomaterials (Fe_3O_4) now a days have been used for various applications such as, biosensing (diagnostics) (Carinelli et al., 2023), drug delivery (Shen et al., 2018), and hyperthermia therapy (Wlodarczyk et al., 2022). Iron oxide can be found in variations of forms in nature. Natural iron oxides, which are mostly in the form of magnetite (Fe_3O_4), hematite (α-Fe_2O_3) and maghemite (γ-Fe_2O_3) can be easily found in iron sand (I Safitri, 2021). Iron sand is essentially black and dark ash in color (Aritonang, 2019).

Figure 2. Iron oxide in their color (al., 2023)

One of the locations in the area of iron sand is located in Tianyar Village, Bali. The potential for iron sand in this region could be affected by the active volcanoes, specially Mounth Agung, which generate lava and ash deposits in a form of cliff or move downslope and provide a source of sediment that is washed into the sea by rivers, particularly during the wet season (Kurnio, 2019). The minerals oxides and hydroxides mineral group, silicates, sulfides, carbonates, and mica group are among those detected in the study area, according to the results of the heavy mineral analysis (Lugra, 2010). Figure 3 shows the spread of magnetite nanomaterials along the Karangasem coastline from south to north. Tianyar village is in the GBT-52 region where it has a fairly high magnetite spread content with a percentage of 18%.

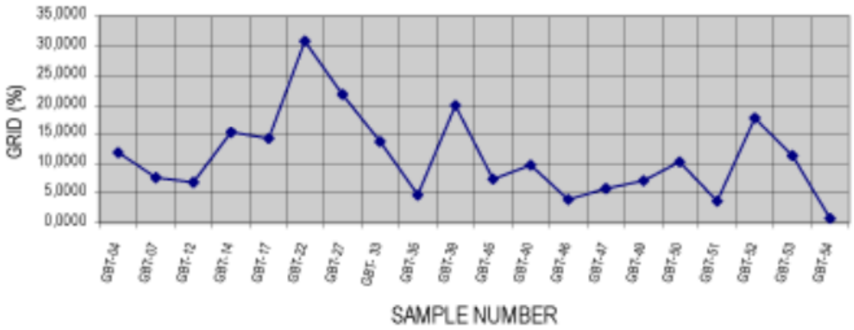

Figure 3. Magnetite Distribution pattern of sediment (from south to the north of Karangasem)(Lugra, 2010).

Iron sand is definitely encouraged to be used for magnetic products due to its high magnetite content. One strategy to enhance the value of iron sand is to use it as a raw material in the synthesis of magnetite (Fe_3O_4) nanomaterials. There are several methods for synthesis Fe_3O_4 nanomaterials, including Co-precipitation (Ba-Abbad et al., 2022), Chemical Vapor Deposition (CVD) (Tyurikova et al., 2020), Sol-Gel (Takai, 2019), ball milling (A. Erwin 2020), and thermal decomposition (Bakr et al., 2020). These synthetic processes aim to alter the characteristics and dimensions of the resulting magnetite particles or crystals (Darminto et al., 2011). The co-precipitation method is the most straightforward synthesis process, requiring few steps and no high temperatures (below 100°C) during the Fe_3O_4 sample synthesis process. Magnetite nanomaterials are obtained by co-precipitation of ferrous and ferric salts in the water medium at different Fe^{+3}/Fe^{2+} ratios, with an ammonia-based strong basic solution at room temperature or above. On the other hand, the co-precipitation synthesis method is a simple and economical method of producing Fe_3O_4 powder from unprocessed iron sand materials (Yusuf et al., 2021).

Co-precipitation synthesis often involves variations in pH, molar ratio (Rahmayanti, 2020), reaction temperature (Nurjanah, 2018), and stirring rate (Rahayu, 2018). pH variations can affect the chemical condition of the solution, alter the kinetics of reactions and particle sedimentation. Some of its impacts include; 1) Particle size, different pH can modulate the rate of nucleation and particle growth, producing particles of different sizes; 2) Morphology, pH can change the shape of particles, such as becoming round or stem-shaped, due to its influence on the crystal growth process; 3) Crystal structure, pH may affect the formation of certain crystal phases and the distribution of crystals within the material (Adhim, 2018). Therefore, the pH adjustment in the synthesis process is a key factor in controlling the physiochemical properties of the magnetite nanomaterials produced.

Nanomaterials can be characterized using various analytical techniques, including spectroscopy, light scattering techniques, X-ray diffraction (XRD) (Alfredo Reyes Villegas et al., 2020), X-ray fluorescence (XRF), scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray dispersive spectrometer (EDS) (Alterary & AlKhamees, 2021), vibrating sample magnetometry (VSM) (Bukit, 2015), as well as a range of other techniques. In the research on Fe_3O_4 nanomaterials, characterization was carried out using XRD and SEM-EDS. XRD characterization is conducted to determine crystal lattice parameters, crystal structure, and crystal size (Cullity, 2014). SEM-EDS (Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy) characterization method is utilized to create microscopic surface images and analyze the elemental makeup of a material in order to investigate its structure and composition.

This research aims to synthesize and characterize Fe_3O_4 nanomaterials from iron sand as the raw material using the co-precipitation method with pH control variations. Mining and excavation, especially sand and rocks, experienced the highest growth in Karangasem district in 2021. However, the main problem for this sector is environmental damage caused by excavations (KARANGASEM, 2022). If iron sand can be converted into magnetic material, it has significant commercial worth (Rusianto). Additionally, this research is expected to contribute to advancements in technology and material research, particularly in studies related to magnetite (Fe_3O_4) minerals and their various applications. Thus, the outcomes of this research hold the potential to create new opportunities for optimizing natural resource utilization and the development of more advanced technologies.

1.2 Problem Limitations

In this research, there are several problem statement and limitations that need to be considered. Firstly, the iron sand used as the primary material for this study is sourced from the Tianyar iron sand deposits. The synthesis process of Fe_3O_4 nanoparticle is conducted using the co-precipitation method, with hydrochloric acid (HCl) as the solvent for iron sand, ammonium hydroxide (NH_4OH) as the precipitating agent, and deionized water as the washing solution. Subsequently, the Fe_3O_4 nanoparticle samples will undergo characterization processes using various analytical techniques, including X-ray Diffraction (XRD) and Scanning Electron Microscopy - Energy Dispersion Spectroscopy (SEM-EDS).

These limitations are crucial as they underscore the source of materials, synthesis method, and characterization techniques employed in this study. The utilization of iron sand from the Tianyar and the application of the co-precipitation method with specific chemical components are integral parts of the research framework. Furthermore, the characterization techniques, encompassing, XRD and SEM-EDS will provide a comprehensive insight into the properties of the generated Fe_3O_4 nanomaterials. All of these limitations support the effective and relevant achievement of the research objectives.

1.3 Problem Formulation

Based on the background presented, the following problem formulations can be summarized as follows.

1. How is the synthesis of Fe_3O_4 nanomaterials derived from Tianyar iron sand using the co-precipitation method with varying pH levels conducted?
2. How is the characterization of Fe_3O_4 nanomaterials derived from Tianyar iron sand using the co-precipitation method with varying pH levels performed?

1.4 Research Objectives

Based on the problem formulations outlined, the objectives to be achieved in this research are as follows.

1. To describe the synthesis of Fe_3O_4 nanomaterials derived from Tianyar iron sand using the co-precipitation method with varying pH levels.
2. To investigate the characteristics of Fe_3O_4 nanomaterials derived from Tianyar iron sand using the co-precipitation method with varying pH levels.

1.5 Benefits of Research

Through this research, the author hopes to provide both theoretical and practical benefits, which are elaborated as follows.

1. Theoretically, this research holds significant potential to encompass various aspects of nanoparticle characterization, especially Fe_3O_4 particles. The utilization of iron sand as a raw material source in the co-precipitation method opens up new opportunities in exploring nanoscale materials with broad applications across various scientific fields and industries. In the context of further research development, the findings from this study can serve as a foundation for subsequent studies that may involve improving synthesis efficiency. Consequently, this research holds the potential for significant impact within the scientific and industrial realms, and it is expected to inspire further research to delve deeper into nanoparticle characterization and the development of innovative synthesis methods.
2. From a practical standpoint, the results of this research have the potential to directly impact the development of technology and industries. The use of Fe_3O_4 particles extracted from iron sand through the co-precipitation method as a raw material can lead to innovations in various industrial sectors. For instance, in the field of information technology, Fe_3O_4 particles have potential applications in data storage, sensor development, and other advanced electronic devices. Moreover, in the medical industry, the potential use of these particles in hyperthermia therapy and drug delivery could improve patient care. This research also carries significant socio-economic implications. Iron sand is an abundant natural resource in these regions, and the extraction of Fe_3O_4 particles can create new sources of income for local communities. This could enhance their quality of life and reduce reliance on traditional livelihoods.