## One-pot synthesis of iron oxide nanoparticles: Effect of stirring rate and reaction time on its physical characteristics

Auni Hamimi Idris<sup>a,b,c</sup> , Che Azurahanim Che Abdullah<sup>a,d,e</sup>, Nor Azah Yusof<sup>b,e</sup>, and Mohd Basyaruddin Abdul Rahman<sup>a,b</sup>

<sup>a</sup>Integrated Chemical BioPhysics Research, Faculty of Science, Universiti Putra Malaysia, Serdang, Selangor, Malaysia; <sup>b</sup>Department of Chemistry, Faculty of Science, Universiti Putra Malaysia, Serdang, Selangor, Malaysia; <sup>c</sup>Faculty of Chemical and Process Engineering Technology, Universiti Malaysia Pahang, Gambang, Pahang, Malaysia; <sup>d</sup>Department of Physics, Faculty of Science, Universiti Putra Malaysia, Serdang, Selangor, Malaysia; <sup>e</sup>Institute of Nanoscience and Nanotechnology, Universiti Putra Malaysia, Serdang, Selangor, Malaysia;

## ABSTRACT

Iron oxide nanoparticles (IONP) have tremendous potential in various applications due to their unique physical and magnetic properties. Controlling synthesis parameters is essential to obtain IONP with desired properties. The influence of stirring rate and reaction time during synthesis of IONP from thermal decomposition of FeOOH on the particle size, crystallinity, and magnetic properties of the IONP were investigated. IONP produced at different reaction times and stirring rates had similar spherical shapes with low polydispersity. Particle size was found to increase from 4.9 nm to 8.6 nm when reaction time increased from 15 to 60 min, with the highest saturation magnetization of 34.3 Am<sup>2</sup>/kg obtained at 60 min. Stirring rates of 400 and 500 rpm produced IONP with better crystallinity and saturation magnetization than 300 and 600 rpm. The properties of IONP can be tuned by selecting appropriate stirring rates and reaction times during synthesis to suit the intended applications.

ARTICLE HISTORY Received 25 July 2021 Accepted 29 March 2022

## **KEYWORDS**

superparamagnetic iron oxide nanoparticles; thermal decomposition; magnetic nanoparticles; stirring rate; one-pot

## Introduction

Iron oxide nanoparticles (IONP) have received considerable attention in the past decade in the biomedical field due to their biocompatibility and magnetic properties. Research is currently being carried out to employ IONP in magnetic resonance imaging (MRI),<sup>[1]</sup> magnetic particle imaging (MPI),<sup>[2]</sup> magnetic hyperthermia,<sup>[3]</sup> and magnetic drug targeting (MDT).<sup>[4]</sup> Three types of iron oxide commonly used in biomedicine are magnetite (Fe<sub>3</sub>O<sub>4</sub>), maghemite ( $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>), and hematite (a-Fe<sub>2</sub>O<sub>3</sub>).<sup>[5]</sup> The magnetic characteristics of IONP can be tuned based on their size and shape to suit the intended purpose. IONP with a core diameter size less than 20 nm typically exhibits superparamagnetism, i.e., the magnetic moments are only present when exposed to a magnetic field and return to zero when the magnetic field is removed,<sup>[6]</sup> making them especially suitable for clinical applications.

Highly crystalline  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> with a size of 14 nm was reported to exhibit excellent heating performance for magnetic hyperthermia.<sup>[7]</sup> However, a recent study by Lappas et al.<sup>[8]</sup> demonstrated that structural defects in particles smaller than 14 nm (~10 nm) are useful for improving heating performance due to changes in their magnetic behavior. In MRI, gadolinium-based chelates are used as contrast agents to enhance the contrast images by shortening either longitudinal relaxation ( $T_1$ ) or longitudinal relaxation ( $T_2$ ), but concern over its toxicity has facilitated development of IONP as potential low-toxic contrast agents. IONP with core sizes below 10 nm demonstrated exceptional quality as positive and also negative contrast agents in lab research.<sup>[9]</sup> IONP with large saturation magnetization enhanced images as negative contrast, yet IONP with low degree of crystallinity and low magnetic moments can be designed as positive contrast agents. Magnetic spin disorder caused by structural defects in ultrasmall IONP was suggested to benefit biomedical applications based on Néel relaxation mechanism, such as magnetic hyperthermia and MPI, but it also could prove useful as  $T_1$  contrast agents.<sup>[10]</sup> Furthermore, IONP with a hydrodynamic size <50 nm is reported to have longer systemic circulation upon intravenous administration due to slower opsonization (adsorption of plasma onto their surface) and clearance from the reticuloendothelial system (RES).<sup>[11]</sup>

The IONP are commonly synthesized using wet chemical routes such as co-precipitation of iron salts, microemulsion, hydrothermal, and thermal decomposition.<sup>[12]</sup> Ligand-assisted synthesis at an elevated temperature generally produces nanoparticles with uniform size distribution due to surface stabilizing ligands during particle formation.<sup>[13]</sup> Hufschmid et al.<sup>[14]</sup> investigated the effect of types of ligands, iron precursor, and the solvent used in IONP synthesis and found that the size and morphology of the IONP are tunable according to these synthesis parameters.