Journal of Alloys and Metallurgical Systems 7 (2024) 100103

Contents lists available at ScienceDirect

Journal of Alloys and Metallurgical Systems

journal homepage: www.journals.elsevier.com/journal-of-alloys-and-metallurgical-systems

Phase structure transformation and growth mechanism for iron oxide nanoparticles synthesized by mechanochemical method: A mini-review

Joseph Ekhebume Ogbezode [a,c,d,*], Nkechi Elizabeth Offia-Kalu [a,e], Abdulhakeem Bello [a,b,f,*], Vitalis Chioh Anye [a], Peter Azikiwe Onwalu [a]

[a] Department of Materials Science and Engineering, African University of Science and Technology, Abuja, Nigeria
[b] Department of Theoretical and Applied Physics, African University of Science and Technology, Abuja, Nigeria
[c] Department of Mechanical Engineering, University of Ibadan, Ibadan, Nigeria
[d] Department of Mechanical Engineering, Edo State University Uzairue, Uzairue, Edo State, Nigeria
[e] Department of Polymer and Textile Engineering, Federal University of Technology, Owerri, Imo State, Nigeria
[f] Centre for Cyber-Physical Food, Energy and Water System (CCP-FEWS), Electrical and Electronic Engineering Science, University of Johannesburg, Johannesburg, South Africa

ARTICLE INFO

Keywords:
Iron oxide nanoparticles
Mechanochemical synthesis
Magnetic properties
Phase transformation
Growth mechanism
Applications

ABSTRACT

The various methods of synthesis of iron oxide nanoparticles (IONPs) have been extensively studied in several works of literature. These methods include physical, chemical, and biological nanosynthesis methods with applications in water filtration, environmental remediation, plant improvements, biomedicines, etc. These nanosynthesis approaches revolve around their mode of application, nanomaterial properties, and characterization mechanisms, while little effort has been made to investigate the effect of nanosynthesis parameters based on phase transformation and growth mechanisms of such IONPs. The parameters, which are physical, chemical, mechanical, mineralogical, and morphological, have proven to have tremendous implications on the magnetic behaviors, crystalline size, degree of crystallinity, lattice stain, and mechanical strength of the synthesized IONPs. Thus, this paper gives an overview of the effect of selected nanosynthesis parameters, potential mechanisms of the phase transformation, nanomaterial characterization, and growth mechanism of IONPs produced via the mechanochemical route. The study also suggests future perspectives on the need for further study on the reduction-oxidation process, reaction kinetics, and growth mechanism as influencing factors that can affect the phase structure transformation and alteration of magnetic properties of mechanochemically synthesized IONPs at various levels for suitability in nanotechnology advancement and applications in various fields.

1. Introduction

1.1. Study background

The mechanochemical method is one of the most efficient conventional green synthesis techniques based on phase structure transformation and growth mechanism in metallic nanoparticles [1]. The solid mineral grinding raw materials for particle size reduction remains the basic principle of mechanical synthesis [2]. Mechanochemical synthesis involves the induction of chemical reactions through mechanical energy input into raw materials [3]. Chemical reactions remain the main difference between the top-down approach and mechanochemical processing of raw materials [4]. It employs the effective mixing of reactants utilizing the ball milling process without or with very little solvents to produce nanoparticles (NPs) of smaller sizes with more surface area and makes it easier for them to be functionalized, all of which are very advantageous for nano-technological applications [5–7]. For instance, different nano-processing techniques have been utilized for the synthesis and characterization of iron oxide nanoparticles (IONPs) [8–10]. However, the problem associated with these conventional IONP synthesis techniques includes the incompatibility of required chemical reagents [11], poor morphological analysis [12], high complexity [13], non-environmental friendly [14,15], high energy requirement [16,17], and time-consuming [18,19]. To mitigate these challenges, a mechanochemical method has proven to be environmentally friendly, time-saving, and requires fewer chemical reagents. A general overview

* Corresponding authors at: Department of Materials Science and Engineering, African University of Science and Technology, Abuja, Nigeria.
E-mail addresses: jogbezode@aust.edu.ng (J.E. Ogbezode), abello@aust.edu.ng (A. Bello).

https://doi.org/10.1016/j.jalmes.2024.100103
Received 25 March 2024; Received in revised form 14 August 2024; Accepted 15 August 2024
Available online 21 August 2024

of the nanocrystalline and phase structure transformation of IONPs from one phase to another based on the growth mechanism and reduction-oxidation reaction by mechanochemical synthesis was discussed.

1.2. Mechanochemical synthesis of iron oxide nanoparticles

In recent years, there has been a lot of study on a novel nano synthesis technique known as "mechanochemical synthesis". This entails the physical activation of solid-state chemical processes by solid-solid displacement reaction during plenary ball milling operation [20,21]. This method has been used to prepare metallic oxides and sulfide nanoparticles [22,23]. It also involves a combination of mechanical milling by distortion and fracture of reactants using a milling machine and a chemical reduction process that occurs within the reactants interface under the influence of external heat at specified temperatures [24,25]. The main benefit of mechanochemical synthesis, unlike other nanoprocessing methods, is that there is no agglomeration throughout the process because it is usually carried out in a solid state (see Fig. 1). This metallic ore ground up to micro- or nano-level by mechanical milling over a period causing particle aggregation and agglomeration by chemical reaction to occur within crystal phases of the grinded nanoparticles after subjection into high energy ball milling operation. This chemical action by mechanical energy induction causes the phase transformation of the chemical properties of the powdered nanoparticles to transit from one stage to another leading to a solid-solid reaction among the particles due to the phase transformation of their microstructure among the concerned metallic nanoparticles.

Moreover, this approach makes it simpler to regulate its overall nanocrystalline size distribution. To fully comprehend the phase transformation and growth mechanism that usually occurs in mechanochemically synthesized IONPs [26], some researchers have attempted to study the effect of different nanoparticle sizes, shapes, magnetic properties, morphology, and phase structure of mechanochemically synthesized IONPs using various chemical reagents and reactants under specified energy conditions [27,28]. Suman et al. [29] examined two different crystalline phases of alpha (α-) and gamma (γ-) Fe_2O_3 structure through the study of their growth mechanism, morphology, and electrical properties under a local electronic structure. Generally, the difference between the crystalline structures (α and γ) of IONPs is depicted in Fig. 2.

Both α and γ atomic structures vary in both crystalline structure and composition. For instance, α-IONPs can be magnetite or hematite with a cubic inverse spinel structure where the crystal lattice entails iron ions, which occupy both tetrahedral (Fe^{3+}) and octahedral (Fe^{2+}) sites. At room temperature, α-IONPs also exhibit magnetic behavior with strong ferrimagnetic tendencies. In contrast, maghemite (γ-Fe_2O_3) is often associated with a partially oxidized spinel structure similar to magnetite, which typically contains Fe^{3+} ions with tetrahedral and octahedral sites. At room temperature, γ-Fe_2O_3 has superparamagnetic material

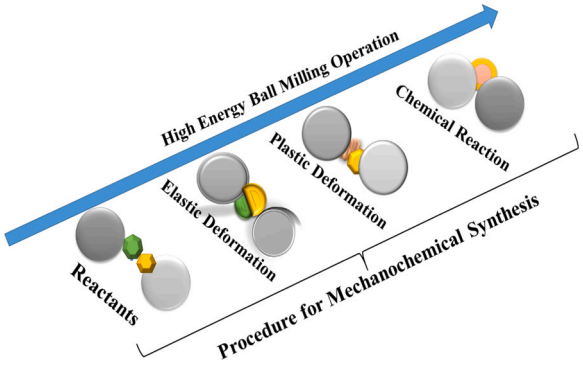

Fig. 1. : Mechanochemical Synthesis by High Energy Ball Milling Operation.

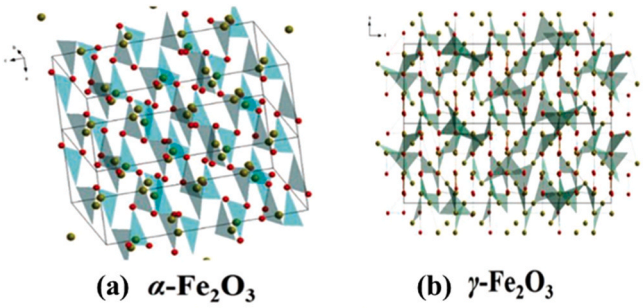

Fig. 2. : Crystal structure of typical IONP **(a)** α-Fe_2O_3, and **(b)** γ- Fe_2O_3 [29].

behavior but exhibits weaker magnetism than magnetite. Thus, the differences in chemical composition, atomic structure, and magnetic properties are major influencing factors that affect the applications of α and γ IONPs in various fields such as biomedical imaging, environmental remediation, and magnetic storage. Fig. 3 depicts the key differences between the atomic structures of α, nanocrystalline, and γ-IONPs synthesized by the mechanochemical method.

Seyedi et al. [30] performed mechanochemical synthesis on α-Fe_2O_3 by a solid-solid reaction between sodium carbonate (Na_2CO_3) and hydrated iron chloride ($FeCl_3.6\ H_2O$). Mechanical induction by the ball milling process was used to transform goethite into hematite nanoparticles in the absence of a reducing agent or additives to produce magnetite nano-crystalline particles, according to Iwasaki et al. [31]. Nanocrystalline transformation of IONPs synthesized by mechanochemical technique has been investigated in few works of literature but IONPs processed via the mechanochemical route could have wider applications in multifunctional materials and technological advancement research compared to other conventional nano-processing routes [32–34]. The vast application of IONPs has recently been sorted due to their particle motion manipulating capability [35], energy dissipation tendencies [36], and imaging contrast because of the influence of the external magnetic field [37,38]. Mechanosynthesis characteristics responsible for the accurate quantification of IONP properties remain a vital component for the effective reproducibility of IONP research with maximum potential in application [39]. On the contrary, several works of literature have attempted to synthesize IONPs using mechanochemical techniques [40–42] but fail to give comprehensive details about the effect of mechanochemical synthesis parameters (i.e., milling time, crystalline size, crystallinity degree, lattice stain, magnetic behavior,

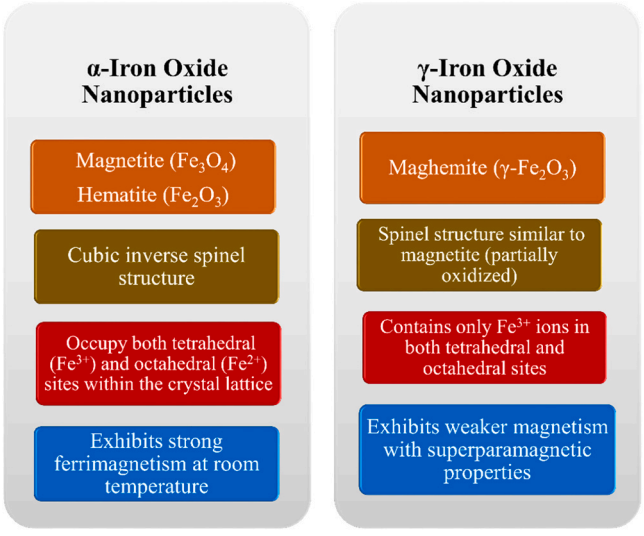

Fig. 3. : Key differences between the atomic structure of α and γ iron oxide nanoparticles.

etc.) on the phase structure transformation of IONPs, causing difficulty in their evaluation and comparison with existing literature [43–46]. Therefore, this paper provides a thorough insight into the influence of the physiochemical parameters on the phase transformation characteristics and growth mechanism of IONPs synthesized by mechanochemical technique. These parameters include magnetic behavior, lattice strain, crystalline size, milling time, grinding speed, etc. The details presented in the paper showed the importance of phase structure characteristics and growth mechanism of IONPs synthesized by mechanochemical method and their most significant applications.

2. Phase transformation of iron oxide nanoparticles by mechanochemical synthesis

The technology of nanocrystalline transformation of IONPs has become a research concern in recent times due to their vast industrial applications. This phase transformation of nanocrystalline particles could be studied through experimental analysis of the particle morphology, phase structure, particle homogeneity, and size distribution which are the main characteristic properties of the transformed nanoparticles which majorly occurred as ferritic and magnetic oxides. Phase transformation of IONPs primarily involves a mechanical milling process by thermal induction [47]. Energy is continually induced in a nanoparticle over a period until it is capable of breaking the chemical bonds between the ionic structures of the base material to produce a different kind of nanoparticle depending on the mode of its industrial application. Gonzalez *et al.* [48], used TEM and XRD analysis to study the nanocrystalline transformation of IONPs from alpha-goethite to alpha-hematite (α-FeOOH to α-Fe_2O_3) through the mechanochemical method. The study was conducted to appreciate the transformation mechanism, formation of IONP phase structure, particle morphology, and crystalline sizes via the topotactic process. This occurs through crystal fragmentation, twin creation, void formation, and loss of hydroxyl group from the goethite nanoparticle. Synthetic goethite was ground at varied milling times up to 104 hrs and phases formed via the high-energy milling process were investigated using the TEM and XRD. Dry-ground mortar was used for the transformation of the synthetic goethite, which is a crystalline powder, into hematite once the oxyhydroxide is powdered at ambient temperature, for a specified milling time. The initial grinding stage reveals the presence of hematite nanoparticles of 10 nm and the final grinding stage at longer hours reiterates a nanoparticle size of about 19 nm. However, few studies have been conducted in the area of the mechanochemical transformation of goethite to hematite NPs especially when it involves the use of natural goethite as the base material. The reason is that the nanocrystalline transformation of goethite majorly entails a topotactic process by dehydration mechanism and thermal energy induction [49]. Literature also attested to the fact that the topotactic process of IONPs occurs by dehydration and thermal induction mechanism either by direct transformation or intermediate superstructure phase formation [50–52]. For instance, the transformation of goethite to hematite can occur as follows, depending on the pathway to nanocrystalline transformation mechanism involved:

α- FeOOH → α- Fe_2O_3 + H_2O or α- FeOOH superstructures → α- Fe_2O_3 -+ H_2O. In similar work, Singh, *et al.* [53], performed a topotactic thermal reduction process on coexisting goethite-magnetite nanoparticles at the atomic scale level under a vacuum annealing temperature of 650 K. The topotactic transformation of the goethite NPs may occur as a partial structural transition from orthorhombic to cubic spinel maghemite (γ - Fe_2O_3) and hematite (α- Fe_2O_3) with a non-stoichiometric nano-structure [54]. The transformation of nano-goethite to nano-hematite is predominately associated with annealing treatment above 500 K which is vital for obtaining anisotropic IONPs [55]. Lemine, [56], performed high-energy ball milling (HEBM) process for the direct transformation of goethite to hematite using the mechanochemical method. α- goethite powder was utilized as the base nanomaterial having undergone a milling process for about 40 hours. Rietveld analysis was used to ascertain the various transformational phases shown between goethite and hematite nanocrystalline structures. XRD, TEM, and vibrating sample magnetometer (VSM) showed the morphology and magnetic properties of the nanocrystalline samples obtained at various milling times. The nanocrystalline transformation process was made possible by the induced mechanochemical effect from the high-energy ball milling (HEBM) process. This nano-processing method is easily produced, time-saving, and environmentally friendly, but only a few such studies have been reported in the literature [57–59]. Therefore, it is expedient to use the mechanochemical method in the production of α-Fe_2O_3 and γ-Fe_2O_3 for the nanocrystalline transformation from α-FeOOH cannot be overemphasized since goethite contains mostly oxyhydroxide ($Fe(OH)_2$) in the soils.

2.1. Potential mechanisms of IONP phase transformation during mechanochemical synthesis

Several potential mechanisms of IONP phase transformation are majorly driven by activation energy during mechanical milling operations which include, the high-energy ball milling effect, solid-solid reaction mechanism due to changes in crystal structure phases, reduction-oxidation reaction among particles, NPs phase selection and kinetics, impact of surface energy and morphological defects of particle (see Fig. 4). These mechanisms' effects result from the high-energy ball milling effect, which involves mechanical energy induced by ball milling high-energy impacts. This effect can lead to deformation, fracturing, or cold welding of the particles. However, the mechanical energy inputs due to ball-milling operation when induced over a period can also lead to local temperature rise despite being conducted at room temperature. Such local temperature increase can result in a localized heating effect due to frictional forces during the milling process thereby influencing the reaction kinetics. As the local temperature rises, a disruption in the solid-solid phases of the base material crystal lattice causes the rearrangement of the milled particle atoms.

The formation of particle metastable phases can also occur under a high-energy milling process. This occurrence is made possible under intense milling conditions which may kinetically favour the milling conditions but affect the stability of the milled particle under equilibrium conditions. The changes in reaction equilibrium automatically result in a mechanochemical process known as a reduction-oxidation reaction due to particle exposure to oxygen in the air or a reactive milling environment during milling. Also, the formation of intermediate reduction-oxidation states of iron ions (i.e. Fe^{2+} and Fe^{3+}) can be transiently formed which represents the final phase transformation of the IONPs. During the phase transformation of IONPs, activation energy barriers are reduced by mechanical energy, enabling the formation of different phases such as Fe_3O_4, γ-Fe_2O_3, or α-Fe_2O_3 depending on the milling conditions. These changes in NP phase structure cause the mechanical energy at the milling operation to alter the particle surface energy, promoting nucleation sites and growth on new phases on particle surfaces. This surface mutilation and whisker growth during the milling process becomes more visible leading to the dislocation of crystal structure and surface defects on the nucleation sites influencing the final NP structure after phase transformation. Thus, understanding the phase transformation mechanism of mechanochemically synthesized IONPs is crucial for parametric studies of their properties because mechanochemistry involves a complex interaction between energy intensities, solid-state reaction, reduction-oxidation, and in-depth kinetics consideration of such NPs. This could no doubt help the synthesized nanoparticles to achieve their desired phase composition, crystallinity, and other properties of the IONPs for more excellent application in various fields of studies (i.e. biomedicine, magnetic materials, catalysis, etc.)

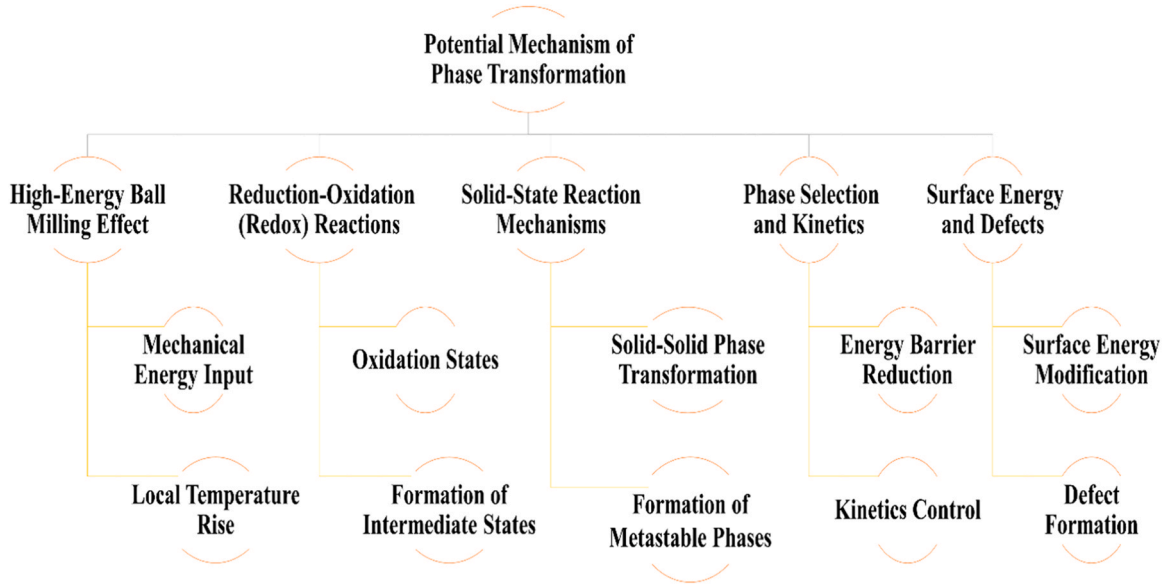

Fig. 4. : Overview of the potential mechanism of phase transformation in IONPs synthesized by the mechanochemical method.

2.2. Influential conditions responsible for IONP phase transformation

Thus, the effect of influencing conditions responsible for the phase transition of IONPs as shown in Fig. 5 has shown a clear interaction with the IONPs processing parameters as shown in Fig. 6 which also does have a major impact on the mechanochemical synthesized IONPs. Table 1 shows the effect of these experimental conditions on IONPs processed by a mechanochemical approach based on the phase transformation characteristics. For clarity, in the scheme for Fig. 6, individual parameters affect the phase structure transformation of IONPs synthesized by the mechanochemical method. The parameters are mechanically intertwined based on the influence of milling time and may decrease with milling speed as the surface area of the grinded IONPs changes with a rise in local temperature. Understanding the relationship between the parameters is crucial for controlling the synthesis process and achieving desired nanoparticle characteristics.

The influencing factors responsible for the phase transformation of IONPs such as mechanical stress, particle coercivity, crystallinity degree, magnetism, etc. are the influential processing conditions that emanate from the parametric activities of the mechanochemical synthesized IONPs. This can be referred to as Influencing nanoprocessing conditions that emanate during the application of parameters affecting IONP phase transformation. For instance, the extent of phase transformation of IONPs during milling is directly linked to an increase in mechanical energy induction for higher phase transformation while the presence of particle agglomeration may lead to undesired phases due to excessive milling time. Increased impact force and energy transfer by mechanical inducement due to higher milling speed may impact kinetic energy on the milled IONPs. An increase in Ball to ball-to-powder ratio implies more mechanical activation energy induced can facilitate phase transformation in the mechanochemical synthesis of IONPs. This operation may lead to cold welding tendencies with a reduction in the effectiveness of the milling process. So, there may be a need to ensure energy balancing between the milling balls and powder mass to avoid excessive nanoparticle size reduction and agglomeration. Considering the type of milling media material based on their hardness, and density, the type of material may also pose a critical effect on IONP phase transformation due to their ability to provide sufficient mechanical energy capable of giving IONPs with desired phase outcome. The presence of atmospheric gases (i.e. air, inert gases, etc.) can easily affect the phase transformation of the milled IONPs. This may emanate from the oxidation-reduction reaction which may favour magnetite and hematite formation depending on the kind of atmospheric conditions involved (oxidation or reduction). Therefore, to achieve stabilized or destabilized phases during the mechanochemical synthesis of IONPs, the selection of the appropriate atmospheric conditions is crucial to achieve the desired phase. In addition, to accelerate or retard IONPs phase transformation, the effect of local temperature rise remains another critical factor that must be checked. Mechanical activated energy can be induced during the milling process which may cause an increase in the local temperature of the NPs which in turn promotes unwanted IONP phases and sintering operation. Thus, a milling operation causing a rise in the local temperature of the milled scale IONPs which can lead to unwanted iron whisker growth and phase transformation must be avoided. Also, the use of smaller particle sizes as starting material during the milling process enhances the easy transition of less stable IONPs to more stable IONPs phase. This phase transition stability with small particles provides NPs with a more uniform and homogenous crystal structure due to higher surface area and energy requirements. The use of external agents (i.e. surfactants, and dispersants) can also be used for phase stabilizing and surface energy alteration in IONP synthesis. These external chemical

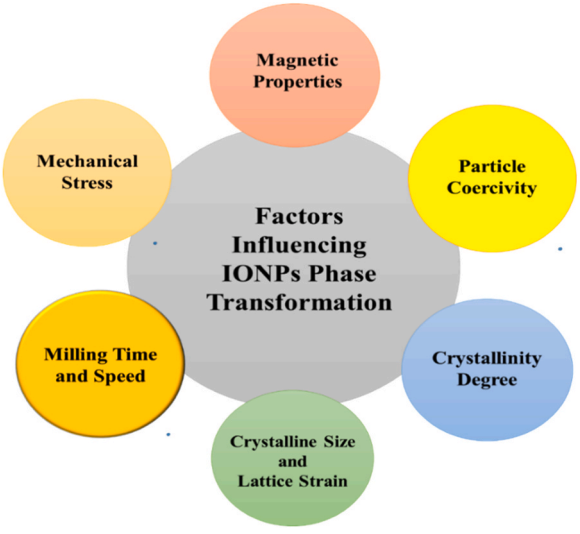

Fig. 5. : Influential nano-processing conditions that emanate during the application of parameters affecting IONP phase transformation.

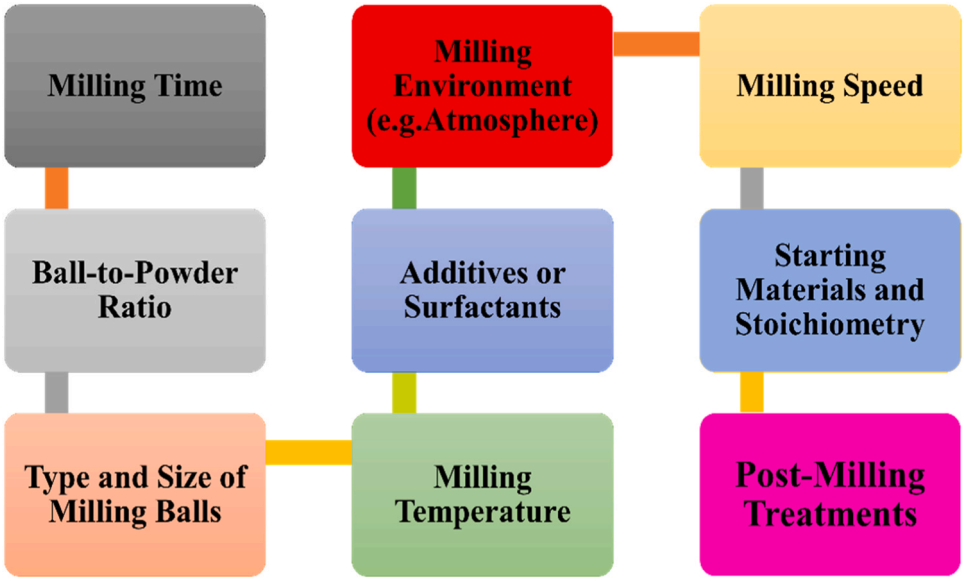

Fig. 6. Parameters affecting phase transformation of IONPs synthesized by the mechanochemical method.

Table 1
Overview of IONPs phase transformation parameters by mechanochemical synthesis

Type of IONPs	Time (hrs)	Temp. (°C)	Speed (rpm)	Size (nm)	Magnetization (emu/g)	Reference
α - Fe_2O_3	5	105	-	4–14	-	Seyedi, *et al.* [30]
Fe_3O_4	7	100	-	15	88	Iwasaki, *et al.* [31]
Fe-O	48	75	-	5–20	80	Kheshtzar, *et al.* [33]
α – FeOOH	40	242	-	53	6.7	Lemine, [56]
Fe_3O_4	48	-	300	33.2	63.68	Can, *et al.* [60]
Fe_3O_4	96	-	200	12	73	De Carvalho, *et al.* [61]
Fe_3O_4 ;	24	300	-	8	46	Badoya, *et al.* [62]
γ - Fe_2O_3				14	68.7	
Fe_3O_4	72	800	-	46	66.4	Lin, *et al.* [63]
Fe_3O_4 ;	12	50	1400	6	5.3	Badoya, *et al.* [64]
γ - Fe_2O_3				12	55.8	

agents alter the landscape of the phase transformation process providing a gangue-way for the direct phase transition process of the mechanochemical synthesis of IONPs.

Thus, the influencing parameters responsible for mechanochemically synthesized IONPs provide a complex interplay and careful optimization of the phase of the transformation and properties of the concerned nanoparticles.

These mechanochemical synthesized NPs emanate from particle size reduction, surface area, increased pore size, etc. Thus, this study also provides an overview of the effect of parameters, magnetic behavior, microstructure, and morphological intricacies on the nanocrystalline transformation of IONPs synthesized by mechanochemical method.

3. Parameters that affect mechanochemically synthesized IONPs during phase transformation

In a mechanochemical synthesis of iron oxides where mechanical energy is used to induce a chemical reaction which later culminates into the phase transformation of such nanomaterials. Many parameters responsible for the phase transformation of IONPs during mechanochemical synthesis are depicted in Fig. 6 including milling time, milling ball type and sizes, ball-to-powder ratio, milling atmosphere, surfactants or additives, milling temperature, base material and stoichiometry, and post-milling operations the phase transformation of the milled particle while greater energy input can be experienced as milling time increases. In mechanical milling, the duration of milling of the grinding process affects the phase structure of the milled nanoparticle thereby promoting phase transformation. The mechanical energy impact per unit of powdered IONPs can then be determined by ball-to-powder. More collision as exerted by the milling balls and powder IONPs causes an increase in phase transformation even as mechanical energy between the milling ball and IONPs powder increases. In addition, energy transfer efficiency and impact strength are also influenced by the size and type of milling balls which can also culminate into faster transformation of phase structure of the IONPs. The environments in which mechanochemical synthesis occurs can also influence the phase transformation of IONPs due to the reduction-oxidation effect of atmospheric gas within the environment. Thus, the use of additives and surfactants can mitigate or modify the reduction-oxidation effect by altering the nanoparticle surface energies or promoting specific chemical reactions and influencing the phase transformation of the IONPs. Although mechanochemical synthesis is usually carried out at room temperature, temperatures might rise locally due to mechanical friction heat. This may also impact phase transitions and reaction kinetics of the IONPs. As the local temperature increases with mechanical energy during milling, initial composition and stoichiometric properties of the iron oxide which is the precursor material as starting material may also influence phases obtained during mechanochemical synthesis. After that, a post-milling treatment (e.g. annealing, alloying, or additional milling steps, etc.) can further alter the energy levels and microstructural properties of the mechanochemically processed IONPs thereby creating a more viable nanomaterial with diverse applications and excellent multifunctional properties. Thus, the optimization and better comprehension of the controlling phases and material structure transformation of

nanosynthesized materials remain crucial in the quest to ascertain their phase composition, crystallinity degree, and material properties of IONPs using the mechanochemical method. Thus, to achieve the required phases of IONPs (i.e. Fe_3O_4, γ-Fe_2O_3, α-Fe_2O_3, etc.) synthesized through the mechanochemical process has become very important for further applications in materials engineering and technological developmental purposes.

3.1. Influence of milling time and speed

Generally, the effect of long milling time and grinding speed can influence the phase structure of mechanochemically synthesized IONPs from different standpoints, such as magnetic property enhancement, structural defect of NP crystal phases, decrease in crystallite sizes, specific phase formation with increased activation energy, and phase purity through homogenization of precursor material (see Fig. 7). For instance, during mechanochemical synthesis, a rise in energy input and prolonged reaction time can favor the formation of different phases of IONPs due to changes in reaction energy levels and precursor phase composition. The size and shape of nanoscale iron oxide can be reduced in smaller NPs, and milling time increases during the high-energy milling process, which may influence the magnetic behavior and optical properties of such IONPs in the long run. Subsequently, the influence of milling time on the purity of the phase formed in mechanochemically synthesized IONPs can be seen. This implies that the formation of single-phase IONPs can be promoted during the extended milling process. Such prolonged mechanical action can foster homogenization of the NPs of the precursor or base material. Also, structural defects can be initiated in the IONP crystal structure due to prolonged milling time. This can give rise to an increase in density as the NPs experience defects and disorder in their crystal structure. The increase in structural defects in mechanochemical IONPs during prolonged milling time can also enhance their magnetic behavior as a result of the optimized formation of new phase structures due to a reduction in NP size. Thus, in the mechanochemical synthesis of IONPs, prolonged milling time typically results in more accurate control over the phase structure, particle size, and characteristics of the end product. The impact of milling time and speed can change based on their particularities, such as the kind of type of milling machine, size of milling balls, type of ball milling material, and environmental impact, as well as the intended usage of the nanoparticles. Therefore, to obtain the appropriate phase structure and characteristics for a given application, experimental milling time adjustment is essential.

The XRD pattern of a mechanochemical reaction between hydrated iron III chloride and sodium trioxocarbonate can be used to produce α – hematite, when milled between 2 and 5 hrs respectively [66]. As depicted in Fig. 7. After milling the powder mixture for 2 and 5 hours, the XRD pattern resembled an amorphous pattern, implying that the product is nanocrystalline iron oxide particles. Iwasaki, *et al.* [67] performed a mechanochemical transformation of α – FeOOH to magnetite nanocrystals. The mechanochemical reduction of highly crystalline superparamagnetic magnetite nanoparticle of size 15 nm was synthesized by direct transformation of α – FeOOH to Fe_3O_4 nanoparticle. The energy induced on the amorphous goethite particle causes nanocrystalline transformation of the goethite to magnetite nanoparticles at different milling times was observed by XRD analysis. An amorphous phase was the initial stage noticed for the goethite NPs, which later crystallized for magnetite to occur even as a single-phase crystal structure was observed at a maximum milling time of 7 hrs. Fig. 7(a) depicts the different phases of the amorphous-crystallized goethite and crystallized-magnetite IONPs at different ball-milling times [68]. Arbian, et al., [69] prepared IONP from hematite powder using the mechanical milling method, the effect of the mechanochemical reduction energy on the base material was investigated at different milling times based on the speed of revolutions by the plenary ball-milling machine. The mechanochemical reduction effect on the process milling time (1, 5, and 10 hrs) and rotational speed (200, 400, and 600 rpm)

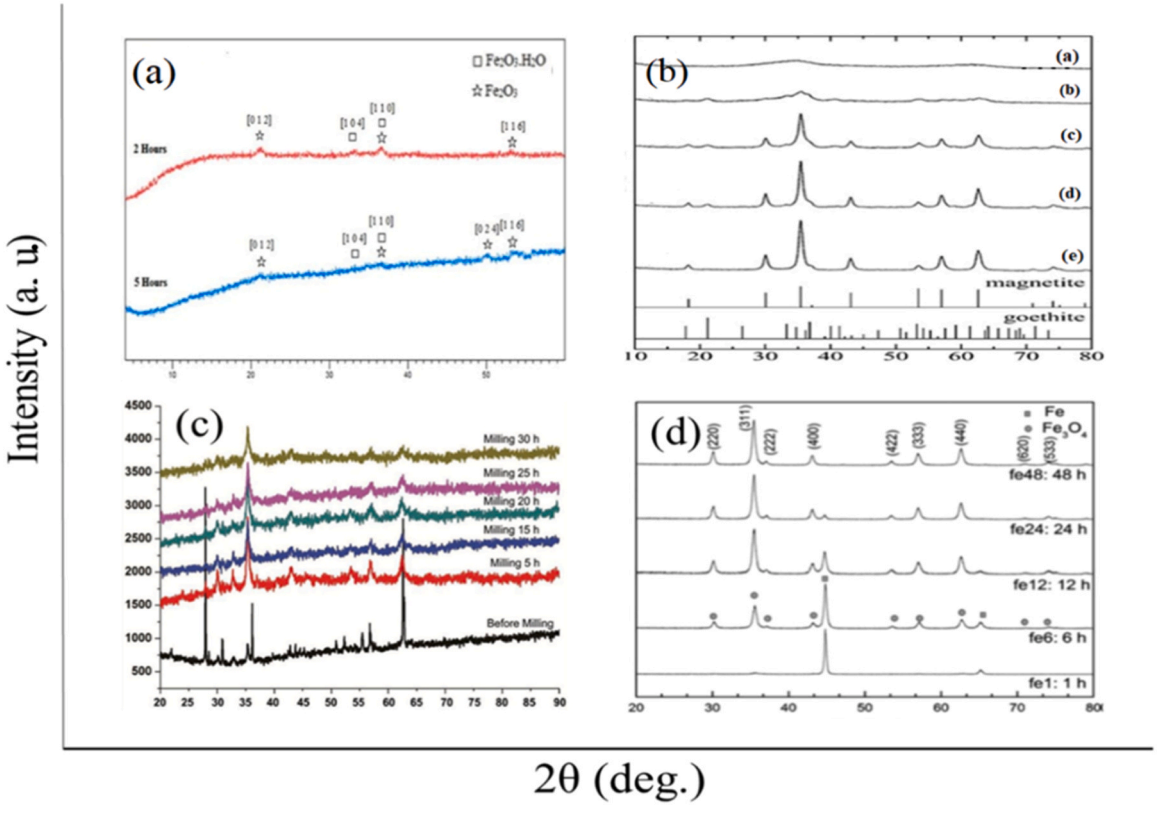

Fig. 7. : XRD patterns of IONPs synthesized by mechanochemical method at a specified milling time (a) α–Fe_2O_3 powder [30] (b) α–FeOOH and Fe_3O_4 [62] (c) α–Fe_2O_3 powder [64] (d) Fe_3O_4 powder [65].

of the ball-milling machine were ascertained and compared to the treated and untreated IONPs at various intensities using XRD analysis. This implies that, for all milled IONP samples, peak intensity is more obvious as milling speed increases from 200 rpm to 600 rpm at a maximum time of 10 hrs. Thus, as the sample phase intensifies, the peak shifts and peak base broadens, while structural distortion of the IONP microstructure shows the formation of the amorphous phase becomes inevitable after mechanochemical reduction has taken place in the milled α – Fe_2O_3 sample. Can, *et al.* [60], performed a mechanochemical synthesis of magnetite NPs under a specified milling time. Magnetite NPs were synthesized from iron sand using the mechanical milling method and the influence of milling time on NP morphology was substantiated via phase transformation mechanism using XRD analysis. The phase transformation of the base material from hematite to metallic iron phase and magnetite to metallic iron phase at various milling times were substantiated using XRD analysis as depicted in Fig. 7(b) and Fig. 7(c). This trend supports the idea that intensive grinding of iron oxide samples for a long milling time causes lattice disorder and crystal structure deformation within the intergranular structure of the IONPs. Fig. 7(d) shows the XRD pattern of the magnetite particle milled at specified times. The XRD pattern depicts the crystalline formation of the IONPs with phase peak shift backward from phase (400) to (311) as milling time hits 12, 24, and 48 hrs. The phase peak shift connotes the transformation of the amorphous to crystalline phase structure of the nanoparticles as earlier envisaged for all magnetic IONPs undergoing mechanochemical synthesis under the HEBM technique. Similarly, phase structure transformation was recorded for other XRD phase change graphs where complete nanocrystalline magnetite formation was observed having attained the set milling times. Thus, higher milling time and speed can also result in a mechanism of high-impact crystal structure breakage which emanates from highly induced mechanical stress and massive strain within the sample crystal lattice [70]. Strong mechanochemical effects on goethite NPs during mechanical milling are time and energy which in turn caused a decrease in peak intensity and Fe – O weakening by the reason of vibration band due to amorphous phase formation as milling time and speed increases [71]. Given this, a more in-depth study on the influence of milling time and speed on IONP synthesis by mechanochemical reduction was done. Extensive consideration of the IONP crystallinity degree, crystallite size, mechanical stress, and lattice strain which are important parameters that affect the iron oxide nanocrystalline transformation and growth mechanism synthesized under HEBM operation was also investigated.

Consequently, the decision to analyze the evolution of IONPs based on nanocrystalline phase transformation is dependent on the need to identify the influence of basic nanoprocessing parameters (i.e. time, speed, size, etc.) on the crystal transformational characteristics of the nanomaterial. Such transformation can be lattice strain, crystal structure, thermal, electrical, and magnetic induction tendencies. For instance, the crystalline transformation of nano-goethite and magnetite under co-existing conditions is quite obtainable using mechanochemical reduction [72]. This may utilize thermal energy induced at 650k further to investigate the topotactic conversion of goethite to hematite nanoparticles. The topotactic formation of IONPs occurs as a result of partial conversion of α – FeOOH to α – Fe_2O_3, further annealing due to thermal reduction which emanates from the local structure evolution of the amorphous α – Fe_2O_3 later metamorphosed in cubic γ – Fe_3O_4. On a general note, transmission electron microscopy (TEM) analysis of the crystallographic structure of IONPs synthesized by the mechanochemical method is shown in Fig. 8. For instance, IONPs synthesized through the mechanochemical route always present nanoparticles with an

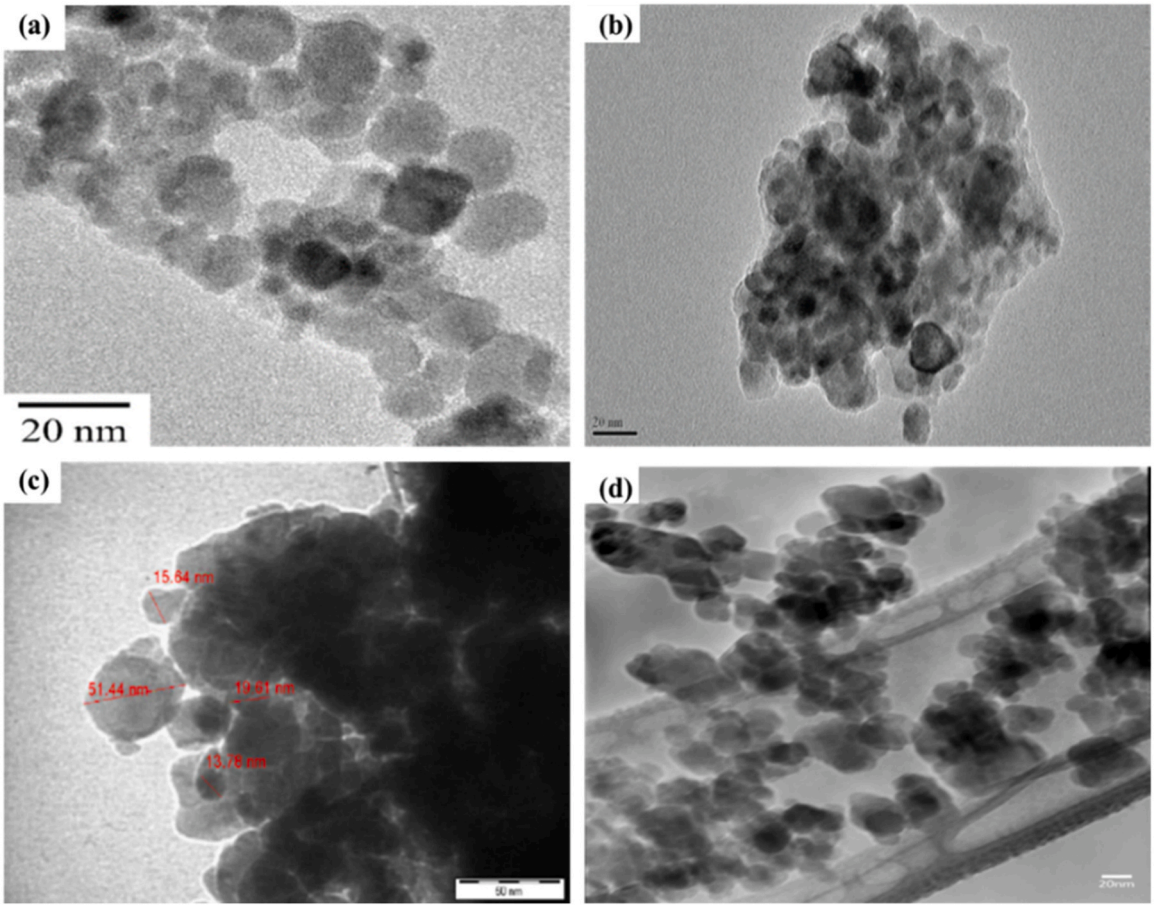

Fig. 8. : TEM analysis of IONP synthesized through mechanochemical route at different milling times **(a)** 7hrs [31] **(b)** 40hrs [56] **(c)** 10hrs [69] **(d)** 48 h [60].

agglomerated phase structure. This becomes imperative because the pronounced particle agglomeration is a major setback for IONPs produced through the mechanical energy-induced milling process. Fig. 8(a) depicts the TEM micrograph of hematite powder ground under plenary ball-milling operation for a milling rate of 40 hrs and a milling speed of 600 rpm. Crystalline IONPs in the 10–60 nm range were obtained with the formation of massive finer particle aggregation. This increases NP aggregation, which becomes possible due to the presence of high surface energy between the IONPs. In addition to their smaller size, the latter effect is primarily caused by the high surface energy resulting from the large concentration of structural flaws caused by intense milling. In Fig. 8(b), the TEM image of α-Fe_2O_3 milled for 40 hours shows an aggregated structure with the nanometric crystallite size of a single crystal structure. For the TEM image of Fe_3O_4 NPs in Fig. 8(c), a mean crystallite size of 30 nm was achieved after a high-energy ball milling process for 48 hours. An obvious agglomeration of particles increases as crystallite size increases with milling time, as roughly spherical structures were distinguished, while this remains a drawback peculiar to mechanically milled IONPs when utilized for application as multifunctional materials for advanced materials research purposes. Fig. 8(d) presents the TEM image of single-phased mechanochemical Fe_3O_4 NPs of homogeneous crystal structure nechanochemically synthesized from α-FeOOH. The IONPs showed the presence of impure phases with decreasing particle size at low crystallinity even as milling time increased. This study may reiterate the agglomeration tendencies of the crystallite size of IONPs and the shape of synthesized IONPs through the mechanochemical route. Although the TEM image of mechanochemically synthesized IONPs can help to ascertain their phase structure, crystallite size, and shape, a topic for further discussion of the details of the mechanochemical reaction mechanism remains necessary. For instance, direct transformation of α-FeOOH to Fe_3O_4 NPs by reduction through continuous reduction by the partial hydrogenation reaction process is possible.

This phase structure evolution has its emancipation from various nanocrystalline parameters which are responsible for the crystal re-orientation within the confines of the grain boundaries of the synthesized IONPs. This re-orientation of crystal lattices of such IONPs can also be susceptible to mechanical milling or mechanochemical synthesis. More explanation on the effect of crystalline size, and lattice strain on phase transformation of mechanochemical synthesized IONPs are further elucidated in Section 3.2.

3.2. Influence of crystalline size and lattice stain

In the mechanochemical synthesis of bulk solid IONPs, the phase transformation of such nanomaterials leads to the development of powerful tools for a wide range of applications owing to their reactivity uniqueness and efficiency. Martinez, *et al.* [73] performed a combined milling and mechanochemical synthesis of magnetic IONPs under different energy sources. The study involves the use of the milling technique as the communication device for the bulk solid IONP, then mechanochemistry reactivity was performed under controlled temperature, electrical impulses, and light irradiation energy conditions to achieve a non-conventional magnetic IONP. This combined nano-synthesis technique which entails thermo-, electro-, and photo-mechanochemistry processes remains the most recent advanced mechanochemistry process of IONPs at solid-state reactivity level. In the mechanosynthesis of superparamagnetic IONPs, microstructure analysis always reveals iron oxide crystals appear in cluster patterns. These nanocrystalline cluster patterns are formed by the multi-crystal Fe-O agglomerates due to magnetic interactions between single crystals [74]. These single and multicrystals of IONPs are major determinants of the superparamagnetic ability of such IONPs [75]. Furthermore, the dense crystal packing of the IONPs also contributes to the magnetic interaction among individual crystals [61]. The crystal sizes of individual IONPs synthesized by the mechanochemical method can be estimated using a Transmission Electron Microscope (TEM) [76,77]. Furthermore, the size distribution and crystal dimensions of IONPs synthesized using the mechanochemical method can be derived from TEM images consistent with the values on the magnetization curves. Magnetic crystals of IONPs are non-interacting and mono-domain based because of the gap between the single crystals [78]. This cluster or agglomeration of single nanocrystals eliminates the mono-domain tendencies of the IONP crystals due to high magnetic interactions within the NP crystal lattice [79]. For mechanochemical nanocrystalline magnetic IONPs, magnetization is high due to possible magnetic interactions within the crystal lattice.

3.3. Influence of coercivity and magnetic properties

Materials science and nanotechnology are very interested in the effects of coercivity and magnetic characteristics on the phase change of IONPs produced mechanochemically. The coercivity of a nanomaterial is a representation of its ability to become magnetized. Thus, IONPs with excellent coercivity properties can have crystalline size, phase structure, and defects within such nanoparticles even as the milling process of the precursor materials via mechanochemical rouse can induce their phase transformation by mechanical energy after a prolonged milling time. Considering the coercivity and magnetic behavior of common phases of IONPs, high magnetization saturation, and lower coercivity are common to Fe_3O_4 NPs since the formation of magnetite can be dependent on its stability under certain milling conditions. For IONPs with γ-Fe_2O_3 phases, an intermediate magnetic behavior, and coercivity are obvious due to their metastable phase, with a high possibility for transformation during prolonged milling into a more stable phase like α-Fe_2O_3. During the prolonged milling phase, the transformation from magnetite to hematite is often experienced as a result of thermodynamic stability. This is possible due to the lower saturation magnetization and higher coercivity of α-Fe_2O_3 compared to magnetite α-Fe_2O_3 and γ-Fe_2O_3. Thus, it is essential to comprehend the relationship between magnetic behavior, coercivity, and phase transformation of mechanochemically synthesized IOMPs for performance optimization in diverse technological and biomedical applications.

For instance, Tsuzuki, *et al.* [80] examined the magnetic property of mono-dispersed γ – Fe_3O_4 through the mechanochemical synthesis in a solid-solid reduction reaction. The mechanism of the IONP phase transformation is more magnetic property dependent than phase-structure based. Investigation of the level of IONP crystalline transformation reveals its superparamagnetic tendencies with a large magneto-anisotropic constant of 6.0×10^6 erg-cm^{-3}. In ferro- and ferri-magnetic materials, the phenomenon called hysteresis can be inevitable due to magnetic field properties around such material [62]. For IONPs, the relationship between the magnetic field strength and magnetic flux density which emanates from such nano-substance is known hysteresis loop [81]. The superparamagnetic properties of IONPs (e.g. maghemite and magnetite) make it possible for the widespread application as spin electronic devices, biosensors, nano-medicine, and magnetic recording devices due to its cubic spinel structure. Shokrollahi, [82], performed a review study on the application of maghemite NPs and synthesis methods based on magnetic properties. For instance, the phase structure of maghemite entails the combination of magnetite and hematite which are of iron oxide family with similar ferrite structure known as ferrimagnetism. Magnetic properties of IONPs can be ascertained using phase structure characteristics in terms of their octahedral inter-granular vacancies, bio-compatibility, and magnetization tendencies [83]. The octahedral vacancies imply their strong tendencies to accommodate certain cations (e.g. Ni^{2+}, Ca^{2+}, Mg^{2+}) into their octahedral site. Lin, *et al.* [63], examined the magnetic properties by mechanochemical reaction using nano-sized Fe_3O_4 of size range 12.5 – 46 nm. The bulk magnetic particles significantly exceed the saturation magnetization property. The increase in saturation magnetization value remains a function of NP crystallite size with a sharp drop in its coercivity property having attained the maximum value. Mechanochemical

synthesis of magnetic IONPs using HEBM was performed by Badoya, *et al.* [84]. The influence of milling materials (e.g. magnetic properties, milling time, chemical stability, etc.) on synthesized IONPs was ascertained. The synthesized IONPs undergo magnetization analysis based on their magnetic field property which was measured via the VSM. The relationship between IONP magnetic field strength and magnetic flux destiny is represented by the hysteresis loop. This property is dependent on the magnetic flux generation capability concerning its changing external magnetic field. Thus, the magnetic behavior of IONPs does have a strong influence on the arrangement of the phase structure based on the method of preparation. Therefore, the literature [85–87] confirmed that IONPs prepared by mechanosynthesis have higher magnetism and lesser magnetic coercivity compared to IONPs prepared by other methods (i.e. sol-gel, sputtering, co-precipitation, etc). Fig. 3 revealed the relation between the magnetization (M) and magnetic field function (H) (i.e. MH curves) of different IONPs synthesized by a mechanochemical method. The saturation magnetization also known as magnetic moment per unit volume is measured at a constant temperature as the applied magnetic field function [88]. The MH curve can also be used to determine saturation magnetization, magnetic diameter distribution coercive field, and remnant magnetization values. For superparamagnetic NPs, the Langevin function and lognormal size distribution are used to analyze the MH curve for the determination of the NP magnetic diameter [64]. In contrast, for non-superparamagnetic NPs, the determination of remanence magnetization and coercivity values are realized using the equilibrium magnetization curve [65]. Thus, for proper verification of the superparamagnetic behavior of IONPs, it is expected that such particles be prevented from physical rotation in alignment with their magnetic dipole in the magnetic field direction. The MH curves allow for IONP magnetic behavioral verification and to determine their saturation magnetization. Fig. 9(a), Fig. 9(b), Fig. 9(c), and Fig. 9(d) depict the magnetic behavior of IONPs synthesized by the mechanochemical method with a saturation magnetization range and coercivity values of 52–66.4 emu/g, 54 – 57 emu/g, 46.02 – 63.68 emu/g, and 42 – 68 emu/g respectively. Also, with coercivity values of 110 Oe, 13 kOe, 11 kOe, and 0.02 T with crystallize sizes of 22.2, 6, and 20, 15 nm respectively. The implication of such variation in the magnetization saturation and coercivity values is entrenched in the method of preparation and the intended application of such IONPs [89–92]. Thus, the degree of magnetic order and preparation temperature of the IONPs remain the fundamental factors that determine their magnetic response in external fields. That is, the spin or orbital energy that emanates from the nanocrystalline surface will define the magnetization value of such IONPs due to the spinel crystalline structure [93]. Furthermore, the determination of the overall magnetization distribution values as shown in Table 1 gives the summary of the chemical and structural stability parameters of recently synthesized IONPs using a mechanochemical technique.

During the characterization of IONPs synthesized via the mechanochemical method for biomedical applications, the measurement of relevant magnetic properties and the method of data analysis cannot be overemphasized. These properties include, coercivity, magnetizing force, saturation magnetization, and flux density are major parameters that can be used to justify the superparamagnetic ability of such IONPs

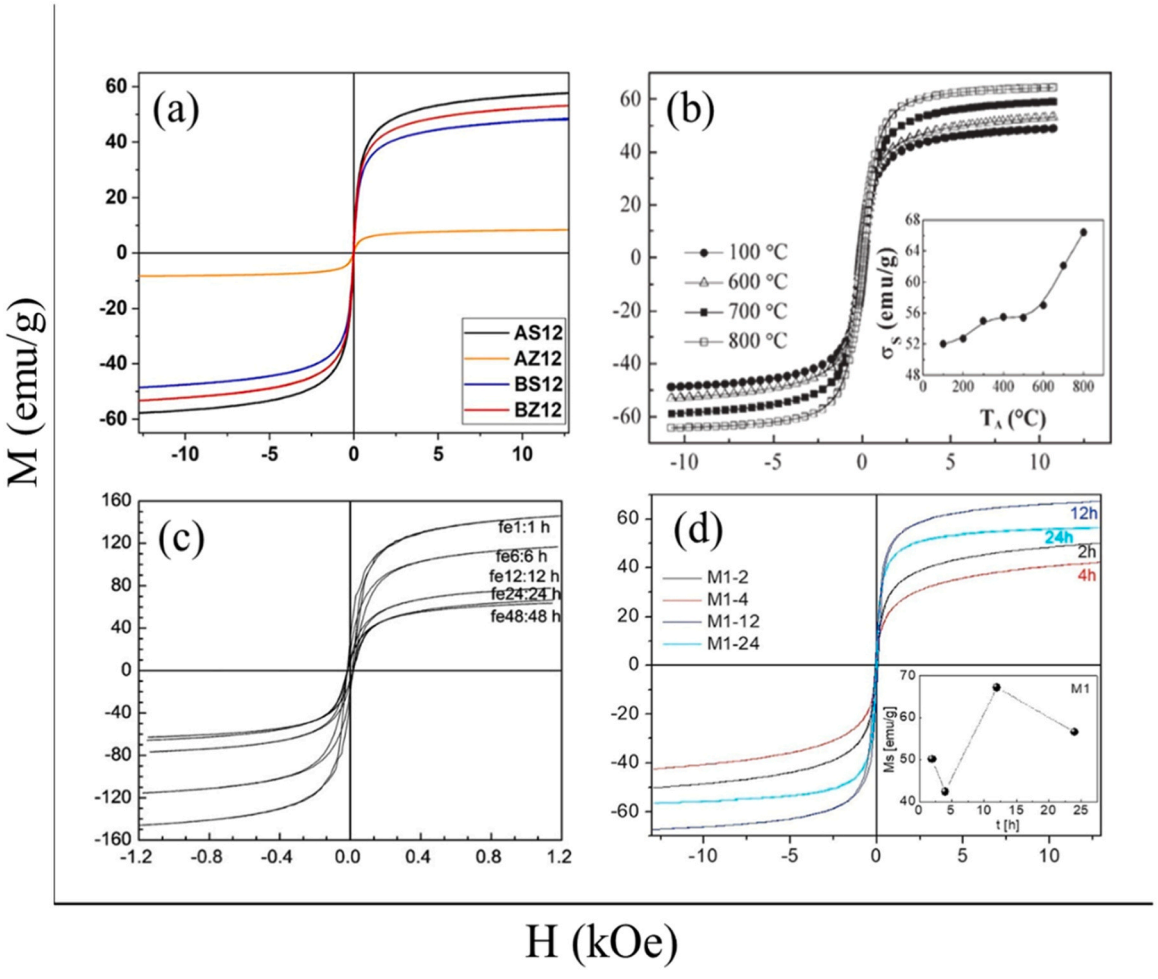

Fig. 9. : Hysteresis Loop Analysis by magnetization (M) as magnetic field function **(a)** Magnetic NPs after 12 hrs [62] **(b)** Magnetite Powder after 1 hr [85] **(c)** Magnetite NPs at 48 hrs [94] **(d)** Magnetite NPs at 48 hrs [95].

[94,95]. The IONPs' response to external force fields depends on the degree of magnetic order and the temperature of preparation [96,97]. Saturation magnetization is also known as the magnetic moment per unit volume of the particle depending on the orbital energy it possesses around its dipoles [98,99]. Maintaining the spin or orbital energy state depends on the crystallite size largeness with multiple domain dipoles separated by a domain wall. For mechanochemical synthesized IONPs for biomedical applications, the crystallite sizes are typically less than 100 nm due to unfavorable orbital energies around the domain walls which may result in single-domain NPs as described by Maldonado-Camargo, *et al.* [100]. The spinel orientation of the magnetic nanocrystalline particles exhibits spinel or inverse spinel structure based on its ferrite nature. This may lead to dipole formation and improve the overall magnetization property of such nanomaterial which is based on metal ion to oxygen distribution in its crystal lattice [101,102]. Also, the strength of synthesized IONPs can be derived using a fitting curve through experimental magnetization properties. Phase transformation of IONP mechanochemical synthesis also possesses the tendency for magneto-crystalline anisotropy [103,104]. The anisotropy property of the IONPs by intrinsic nature could be induced by external processes, magneto-crystalline property, exchange anisotropy, and crystalline shape. IONPs generally exhibit ferri- and ferromagnetic magnetic behavior known as hysteresis [105,106]. This involves the ability of magnetocrystalline anisotropy to form a desired crystallographic alignment pattern. Thus, the rate of alignment is time-dependent while the direction of the magnetic field is temperature-dependent [107,108]. To fully comprehend the benefits of magnetic behavior as an important factor in the synthesis and characterization of IONPs, more investigation on the influence of temperature, and rate of nanocrystalline alignment, within the magnetic anisotropic energy barrier must be ascertained. Also, magnetocrystalline anisotropy which is an important factor for determining the effect of temperature (thermal energy) and rate of magnetic force field on the superparamagnetic ability of the IONPs in terms of their zero-field and magnetic susceptibility should be further investigated.

3.4. *Influence of crystallinity degree and mechanical stress*

In the production of nanostructured particles by mechanochemical synthesis, the energy induced into the metallic oxide samples is due to the effect of mechanical stress which is targeted towards the morphological characteristics of such nano-based material [109]. Among these parameters is the degree of crystallinity which involves the IONP transformation parameter. The degree of crystallinity can simply be referred to as the extent of mechanochemical reduction exhibited on the nano-structure of a metallic oxide at a specified milling time and milling speed [110,111]. This can also be termed as the extent of structural distortion of the crystal lattice such IONPs due to the mechanochemical effect induced in the form of stress energy which causes a decrease in the degree of crystallinity while induced stress energy increases. This also causes a soft agglomeration within NP aggregation as milling time and milling speed increase [112–114]. This growth mechanism and phase transformation of IONPs may also emanate when prepared under intensive milling conditions, due to the agglomeration of micropores because it is susceptible to other inorganic elements (i.e. nitrogen) which may lead to the breakdown of larger metallic oxides within its crystal structure into smaller bits. Thus, the induced stress energy under mechanochemical reduction of IONPs may also result in a gradual decrease in crystallinity when treating IONPs of larger particle size at specified induced stress energy. Thus, the conversion of amorphous to crystalline IONP could be made possible using the mechanochemical reduction process via nano-crystalline transformation of IONPs under induced stress energy with a gradual decrease in the degree of crystallinity.

4. Conclusion and future perspectives

The need to synthesize IONPs that are less energy-intensive, environmentally friendly, and cost-effective cannot be overemphasized. Major preparation intricacies and challenges attached to IONPs produced by other conventional nanofabrication techniques have shortened the extent of their effective applications in real-time situations. These challenges include poor nanomaterial processing techniques, high costs of preparation, limited ways of application, poor in-depth analysis of IONP phase transformation, and so on. This IONP phase transformation remains possible under high-energy milling operations when synthesized using mechanical milling and mechanochemical methods. Other areas of nanoprocessing methods are biological, chemical, and physiochemical. The latter nanosynthesis method has proven to be more environmentally friendly but very expensive. Phase transformation parameters (i.e., magnetic diameter distribution, saturation magnetization, coercive field, and saturation magnetization) in IONPs synthesized are responsible for their maximal application in various fields of study. This study concludes that:

- The IONPs prepared by mechanochemical synthesis remain a foremost combination of physical and chemical methods in nanofabrication techniques with vast applications in electromagnetic devices, magnetic resonance imaging (MRI), biomedicine, water filtration, and environmental remediation purposes. Its vast application capabilities resulted from purification tendencies, cost-effectiveness, eco-friendliness, and time-saving tendencies.
- The preparation of superparamagnetic IONPs is often performed by the mechanochemical method at room temperature through a combination of aqueous suspension and ball milling treatment. Mechanochemical synthesis remains apparent in the growth mechanism and phase structure transformation of superparamagnetic IONPs due to its mitigation effect on the reduction of toxic agents, organic substances, and surfactants around the crystal lattice of the base material.
- The study of phase transformation and growth mechanisms of IONPs produced by mechanochemical synthesis can be useful for surface modification, particle compounding, and environmentally friendly nanofabrication techniques for multifunctional materials.
- The impact of milling processing factors in the mechanochemical synthesis of IONPs may change based on their particularities, which include, the type of milling machine, milling ball sizes, ball milling material, and environmental factors, as well as the intended usability of the nanoparticles. Therefore, to obtain the appropriate phase structure and characteristics for a given application, experimental milling time adjustment is essential.
- The quest to perform the nanocrystalline process via a green synthesis approach cannot be overemphasized. The need for an in-depth study on the potential mechanism of phase transformation and characteristics of IONPs produced by mechanochemical synthesis is quite imperative for proper verification of the magnetic behavior and phase structure of mechanochemically synthesized IONPs.
- Optimization of IONPs for better comprehension of their controlling phases and crystal structure transformation of such synthesized nanomaterials remain crucial in the quest to ascertain their phase composition, crystallinity degree, and material properties of such NPs using the mechanochemical method. Therefore, achieving those required IONP phases (i.e. Fe_3O_4, γ-Fe_2O_3, α-Fe_2O_3, etc.) through mechanochemical synthesis has become very imperative for their future applications in smart and multifunctional materials for advanced engineering and technological developmental purposes.
- Also, the magnetic behavior characteristics of IONPs prepared by a mechanochemical approach are dependent on the typical measurements of the IONP sample, which are measured as the applied magnetic field function under specified isothermal conditions.

- The magnetic properties of magnetic IONPs can be ascertained using the MH curves to fully substantiate their magnetic diameter distribution, saturation magnetization, coercive field, and remanent magnetization.
- To fully understand the superparamagnetic properties of IONPs using mechanochemical synthesis, further investigation on their nanocrystalline rate alignment within the magnetic anisotropic energy barrier should be ascertained. This magnetocrystalline anisotropy can be determined based on the rational relationship between the orbital energy barriers and the magnetic field function of the NPs based on zero magnetic susceptibility.
- Further analysis of the growth mechanism and phase transformation of IONPs synthesized by mechanochemical reactions remains very sacrosanct as far as the mechanical, morphological, and magnetic properties of the transformation of IONPs are concerned.
- Other factors responsible for the transformation of the IONP phase structure of IONPs, the growth mechanism that can induce mechanical stress, and the degree of crystallinity have proven to produce an effective conversion of IONPs from the amorphous to the crystalline phase. The increase in IONP crystallinity degree is made possible when produced under the mechanochemical reduction process at specified induced mechanical stress.

Funding declaration

This work was supported by the Partnership for Applied Science and Engineering Technology (PASET) and Research Scholarship Innovation Fund (RSIF) with Grant number: B8501E21184.

Abbreviations

EDS	Energy dispersive spectroscopy
DMSO	Dimethyl sulphoxide
HEBM	High-energy ball milling
HR-TEM	High-resolution transmission electron microscope
FTIR	Fourier transforms infrared
MRI	Magnetic resonance imaging
NPs	Nanoparticles
IONPs	Iron oxide nanoparticles
nZVI	Nanoscale zero-valent iron
SEM	Scanning electron microscope
SFMION	Surface-functionalized magnetic iron oxide nanoparticles
SPION	Superparamagnetic iron oxide nanoparticles
TEM	Transmission electron microscope
TGA	Thermogravimetric analysis
TG-DTA	Thermogrametric/Thermal differential analysis
XRD	X-ray diffraction
XRF	X-ray fluorescence
VSM	Vibrating sample magnetometer
ZVMI	Zero valent metallic iron

CRediT authorship contribution statement

Vitalis Chioh Anye: Validation, Supervision, Project administration. **Abdulhakeem Bello:** Writing – review & editing, Supervision, Project administration, Formal analysis, Conceptualization. **Nkechi Elizabeth Offia-Kalu:** Writing – review & editing, Validation. **JOSEPH EKHEBUME Ogbezode:** Writing – original draft, Visualization, Validation, Resources, Methodology, Investigation, Funding acquisition. **Peter Azikiwe Onwualu:** Validation, Project administration.

Conflict of interest

The authors have no competing interests to declare that are relevant to the content of this article.

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