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Fe_3O_4@PSC nanoparticle clusters with enhanced magnetic properties prepared by alternating-current magnetic field assisted co-precipitation

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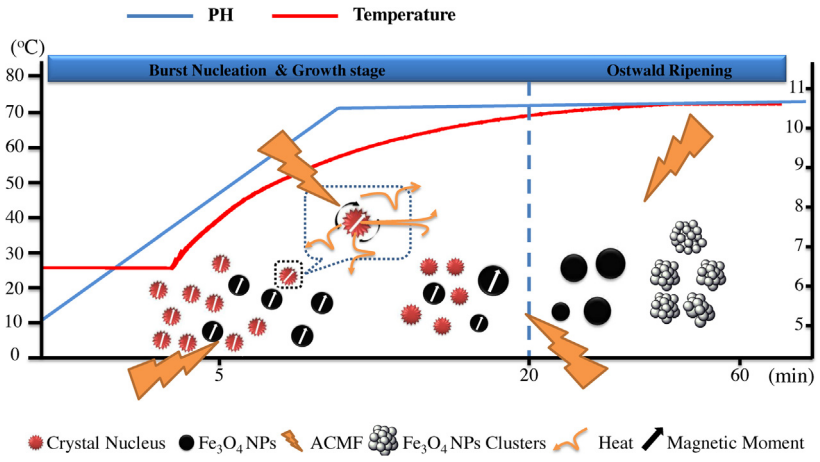
HIGHLIGHTS

- Fe_3O_4 nanoparticle clusters with enhanced magnetic properties were prepared successfully by ACMF-assisted co-precipitation.
- The magnetic effect as well as heat effect of ACMF on Fe_3O_4 nanoparticles plays a key role in the enhancement of magnetic properties during the whole synthesis process.
- This strategy might hold promise in preparation of high performance magnetic nanomaterials in the future.

GRAPHICAL ABSTRACT



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ABSTRACT

Field-assisted synthesis has been one of the common strategies of nanoparticles preparation with enhanced properties. However, most researches focused on exploring the effects of monofunctional static field on the structure and properties of synthetic nanomaterials, few researchers have applied fields with period variation as assistance. Combining medium alternating current magnetic field with most widely used method traditional chemical co-precipitation held potential in preparation high quality Fe_3O_4 nanoparticle. In this study, Fe_3O_4 nanoparticle clusters were prepared in alternating-current magnetic field (ACMF) by co-precipitation principle, structural and magnetic properties were also characterized. Results demonstrated that Fe_3O_4 nanoparticle clusters prepared by co-precipitation heated in ACMF indicated a better heat production under ACMF, comparing with Fe_3O_4 nanoparticle clusters with similar size and distribution prepared by classic co-precipitation. It might be due to the magnetic effects induced by ACMF that Fe_3O_4 nanoparticles tend to grow along the magnetization direction. This technology might hold promise in preparation high performance magnetic nanomaterial in the future.

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1. Introduction

The effects of applied static field on the morphologies and properties of nanomaterial during the synthesis have been one of the hot spots for a long time [1]. Numerous researches paid attentions to the influence of static field on the dynamics and thermodynamics during the synthesis [2,3]. In addition, some similarly concentrated on rapid synthesis of magnetic nanoparticles on the assistance of time-varing field with high frequency, for example the microwave field [4,5]. Among the various fields, alternating-current magnetic fields (ACMF), especially ACMF with low or medium frequency, have been wildly used in biomedical engineering areas [6]. Many works about magnetic inductive hyperthermia [7] and drug controlled release based on ACMF have been reported [8–10], which confirm that human body is tolerated by low or medium frequency ACMF. On account of this safety, nanomaterial with good biocompability and stability in body use for instance iron oxide nanoparticles could be prepared with assist of ACMF, yet few appeared.

As one of the most wildly used magnetic nanomaterial in biomedical applications, iron oxide nanoparticles have been drawn attentions in many applications as bioseparation agent [11–13], drug carrier [14,15] and so on. The major advantages of well modified iron oxide nanoparticles is the good biocompability and stability. There are considerable iron oxide nanoparticles being approved and for clinical research. Ferumoxytol, a novel type of iron oxide nanoparticles, have been proved by USA Food and Drug Administration (FDA) as drugs for iron supplementation [6]. Besides, iron oxide nanoparticles with unique structures and characterizations could be fabricated with the assistance of applied static magnetic field [16,17].

As we all know, chemical co-precipitation process can well be explained by Lamer mechanism that a short burst of nucleation from a supersaturated solution and the slow growth of particles without any significant additional nucleation. And based on Brown relaxation and Néel relaxation theory [28], magnetic nanoparticles can produce heat accompanied with relaxation process of nanoparticles, in this procedure magnetic nanoparticles is magnetized and the magnetic moment is gradually arranged. In previous study [6], we have observed that the tiny particles after the initial nucleation can generate heat and increase reaction system temperature in the ACMF produced by moderate-radiofrequency heating machine, and have successfully prepared ferumoxytol, one kind of magnetic iron oxide nanomedicine with regular size and good magnetism.

In the meantime, we also found that PSC shows a prominent intervention effect on the nucleation and growth during the formation of iron oxide nanoparticles [18], so the iron core and hydrodynamic size of nanoparticle will be constrained as well as the magnetism, causing this nanoparticle inadequate in many applications *e.g.* magnetic hyperthermia. Herein, an alternative synthetic strategy developed for large size and high magnetic properties was finished. We aimed at adding PSC at the end of aging process in ACMF to alleviate the block effect, Fe_3O_4 nanoparticle clusters with better magnetic properties could be formed instead of monodispersed nanoparticle. And the result showed comparing to nanoparticle clusters with similar size and dispersivity prepared by classic co-precipitation, this Fe_3O_4@PSC nanoparticle clusters indeed demonstrated higher saturation intensity and a better performance in heat generation in ACMF, which is great potential in magnetic hyperthermia.

2. Experimental

2.1. Materials and reagents

$FeCl_3{\cdot}6H_2O$ and $FeCl_2{\cdot}4H_2O$ were purchased from Aladdin Chemical Reagent Company (Shanghai, China), 28% aqueous ammonia was from Lingfeng Chemical Reagent Company (Shanghai, China), Polydextrose sorbitol carboxyl methyl ether (PSC) was prepared from Jiangsu Key Laboratory for Biomaterials and Devices. All the chemical reagents were AR grade.

2.2. Fabrication of Fe_3O_4 nanoparticle clusters

To explore the difference in magnetic property between the nanoparticles clusters prepared by ACMF and those by oil bath, we firstly prepared samples by ordinary oil bath (70 °C), and the amount of each material will be consistent with those of prepared in ACMF. we established an ordinary co-precipitation method after screening important factors in the reaction, confirmed each concrete parameter such as Fe^{3+}/Fe^{2+} ratio, the initial concentration of PSC and other steps followed by the traditional chemical co-precipitation pricinple. Then, Fe3O4 nanoparticle clusters were fabricated in the ACMF and characterized for comparison with that prepared in classic method.

In brief, 0.74 mM of $FeCl_3{\cdot}6H_2O$ and 0.5 mM of $FeCl_2{\cdot}4H_2O$ were dissolved thoroughly in 5 ml of water in the round bottom plastic tube immobilized in the ACMF induction coil with a stirrer made of polytetrafluoroethylene and continuous nitrogen protection. The stirring speed was 600 rpm. Then 1 ml NH_4OH aqueous solution (5 mM/mL) was added dropwise to the reactor with the operation of the moderate radio frequency heating machine (Shuangping SPG-10-II, 340 KHz, 20 A, China) until the whole mixture became black, indicating the formation of Fe_3O_4 nanoparticles. Then the reaction system was heated to 70 °C and the output electric current of device was adjusted at 15A to maintain temperature, then aged for 40 min before the addition of 2 ml PSC aqueous solution (100 mg/mL). After that, the mixture was aged for another 15 min for unchanged temperature and output electric current. After it was cooled to room temperature, the nanoparticles was washed twice with ultrapure water to remove the redundant PSC, Fe^{2+},Fe^{3+},NH_4^+ and Cl^-. The final products were collected through magnetic separation and dispersed in water. Moreover, another nanoparticle was obtained by ordinary co-precipitation method, and the materials and preparation process was the same with above steps except using oil bath as heat source.

2.3. Characterizations of Fe_3O_4 nanoparticle clusters

The morphology of the samples was examined by transmission electron microscope (TEM)(JEOL JEM-2100, Japan), The distribution state of nanoparticles clusters was characterized by Scanning electron microscope (SEM)(Zeiss, Ultra Plus, Germany), hydrodynamic diameter of the samples was measured by dynamic light scattering (DLS) (Malvern ZS-90, England), the crystal phase of samples was examined by x-ray powder diffraction with Cu Kα radiation on a X-ray diffractometer (Thermo X'TRA, USA) at room temperature. The magnetic properties (*M-H* curve) of the samples were measured by Vibrating sample magnetometer (VSM) (Lakeshore 7407, USA). SAR evaluation of the sample dispersion (1 ml, 1 mg/mL Fe) was performed using an ACMF generator (Shuangping SPG-06-II, China) with the applied frequency was 390 KHz. The infrared thermal images of the samples was recorded by an Infrared Thermal Imager (Fluke Ti32, USA).

3. Results and discussion

The size and morphology will greatly affect the properties of nanomaterial [19,20]. Researches indicated that the single particle size would affect the heat generating under alternating magnetic field with different frequency [21]. TEM images indicated that by controlling the synthetic conditions, the single particle size of Fe_3O_4@PSC nanoparticle clusters prepared by alternating magnetic

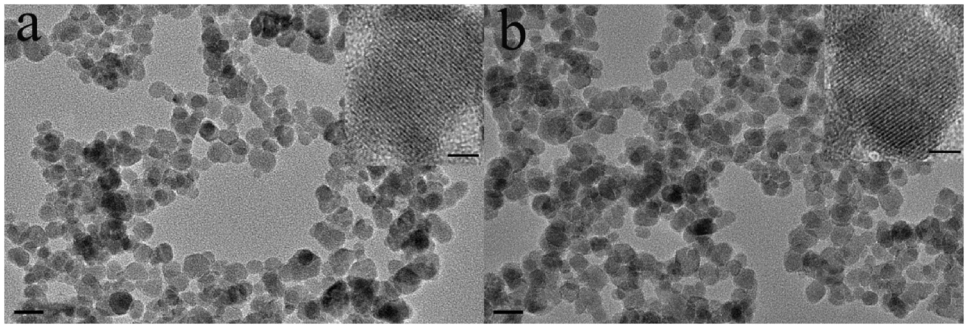


Fig. 1. TEM images of magnetic nanoparticle clusters prepared by alternating magnetic field assisted co-precipitation (a) and classic co-precipitation (b). Scale bar: 20 nm. HRTEM images of a single nanoparticle prepared by alternating magnetic field assisted co-precipitation (a) and classic co-precipitation (b). Scale bar: 5 nm.

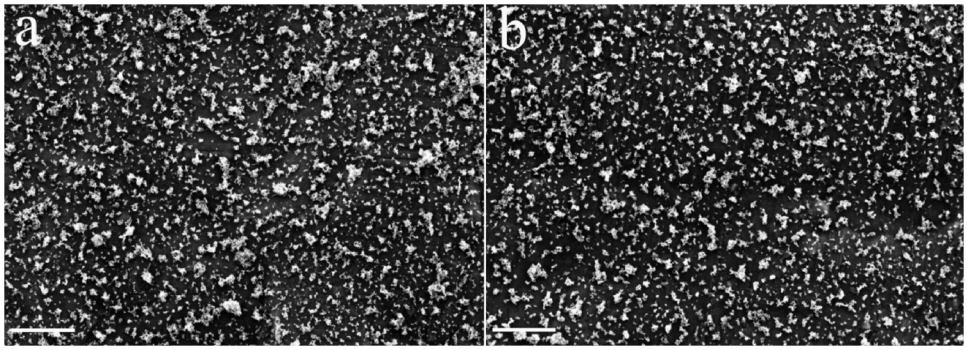


Fig. 2. SEM images of magnetic nanoparticle clusters prepared by alternating magnetic field assisted co-precipitation (a) and classic co-precipitation (b). Scale bar: 1 μm.

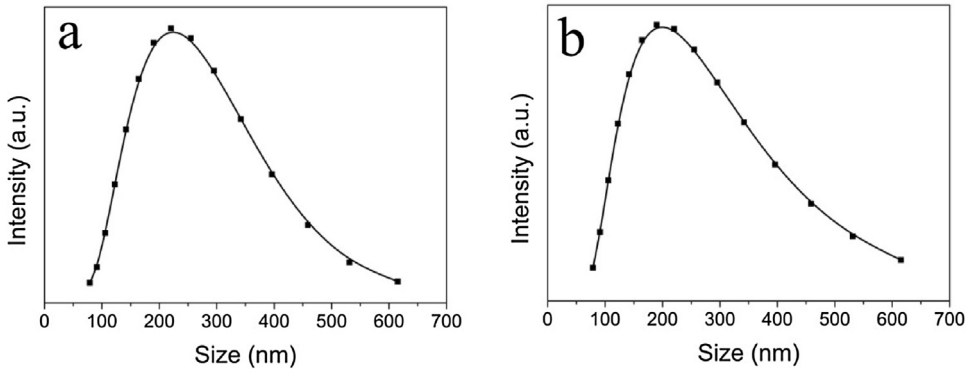


Fig. 3. DLS images of magnetic nanoparticle clusters prepared by alternating magnetic field assisted co-precipitation (a) and classic co-precipitation (b).

field assisted co-precipitation was 12.7 ± 1.2 nm and the single particle size of Fe_3O_4@PSC nanoparticle clusters prepared by classic co-precipitation was 12.5 ± 1.6 nm (Fig. 1), which were both close to the optimal size for hyperthermia under ACMF with medium frequency [21]. The high resolution TEM (HRTEM) images of a single nanoparticle prepared by ACMF assisted co-precipitation and classic co-precipitation showed the lattice spacing of 4.83 Å, which corresponded with the (111) plane of the cubic structure of Fe_3O_4. The selected area electron diffraction (SAED) was shown in Fig. S1. The presence of clear rings in Fig. S1 shows that the samples are all polycrystalline. Comparing to ferumoxytol synthesized in our previous work, the average size clearly increased. It was closely associated with the absence of PSC during the nucleation and growth of Fe_3O_4, which indulged the whole reaction process and leaded to the larger nanoparticles clusters [22].

Due to the high surface energy of unmodified magnetic nanoparticle prepared by co-precipitation, single nanoparticle intended to form large clusters. SEM images demonstrated that both samples were clusters composed of small nanoparticles (Fig. 2). The morphology of these nanoparticle clusters showed no obvious differences.

Hydrodynamic diameter obtained by DLS matched with the SEM and TEM results (Fig. 3). The hydrodynamic diameter of Fe_3O_4@PSC nanoparticle clusters prepared by alternating magnetic field assisted co-precipitation was 208 ± 58 nm and the hydrodynamic radius of Fe_3O_4@PSC nanoparticle clusters prepared by classic co-precipitation was 201 ± 49 nm. All these characterizations supported that the structures and morphologies of these magnetic nanoparticle clusters prepared by alternating magnetic field assisted co-precipitation and classic co-precipitation were of no great difference.

The crystal formation and degree of crystallinity are keys factors which influence saturation magnetization and heat generation properties of magnetic nanomaterial [23]. The peaks at 18.3°, 30.2°, 35.6°, 43.1°, 53.3°, 57.0°, 62.4° and 75.0° in XRD spectrum represented Fe_3O_4 crystal plane 111, 220, 311, 400, 422, 511, 440, 622

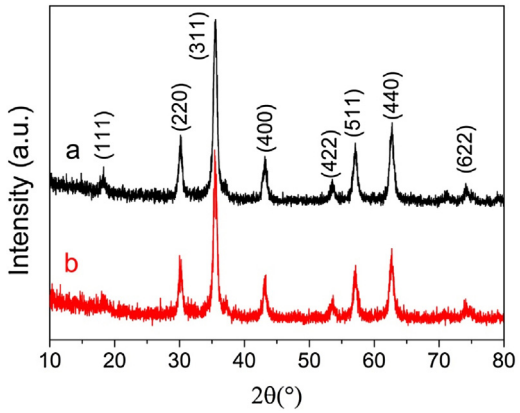


Fig. 4. XRD spectrum of magnetic nanoparticle clusters prepared by alternating magnetic field assisted co-precipitation (a) and classic co-precipitation (b).

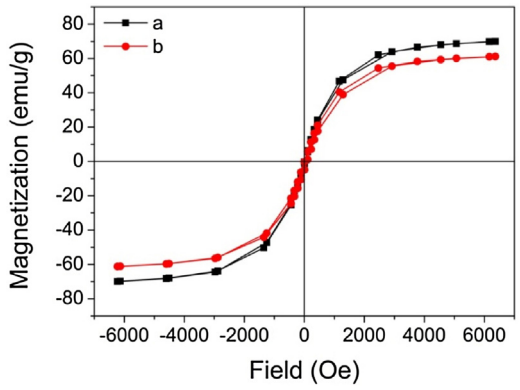


Fig. 5. VSM spectrum of magnetic nanoparticle clusters prepared by alternating magnetic field assisted co-precipitation (a) and classic co-precipitation (b).

respectively (Fig. 4). XRD results showed that samples prepared by ACMF assisted co-precipitation and classic co-precipitation were both Fe_3O_4 nanoparticles. The crystal transformation from Fe_3O_4 to Fe_2O_3 could lead to the reduction of Saturation Magnetization, which would consequently gave rise to the reduction of magnetic heat production capacity [24,25]. Thus compared with Fe_2O_3 nanoparticles (*e.g.* ferumoxytol) prepared in our previous work, the Fe_3O_4 nanoparticles clusters prepared in this study have better advantage in magnetic heat production capacity.

Saturation magnetization also plays an important role in altering the magnetothermal capability of nanomaterial. VSM was used to evaluate the magnetic properties of Fe_3O_4@PSC nanoparticle clusters prepared by ACMF assisted co-precipitation and classic co-precipitation. Results indicated that the saturation intensity of Fe_3O_4@PSC nanoparticle clusters prepared by ACMF assisted co-precipitation was 69.2 emu/g, which was slightly larger than the saturation intensity (61.3 emu/g) of Fe_3O_4@PSC nanoparticle clusters prepared by classic co-precipitation with identical size (Fig. 5). Furthermore, the coercive force and residual magnetism of these nanomaterial were different. The coercive force and residual magnetism of Fe_3O_4@PSC nanoparticle clusters prepared by classic co-precipitation was circa 6 Oe and 0.3 emu/g respectively (Fig. 6a). However, the coercive force and residual magnetism of Fe_3O_4@PSC nanoparticle clusters prepared by alternating magnetic field assisted co-precipitation were circa thrice than those of Fe_3O_4@PSC nanoparticle clusters prepared by classic co-precipitation, which were 20 Oe and 1 emu/g accordingly (Fig. 6b). Statistic results based on ten different batch of Fe_3O_4@PSC nanoparticle clusters prepared by classic co-precipitation and alternating magnetic field assisted co-precipitation showed that the difference of magnetization between Fe_3O_4@PSC nanoparticle clusters prepared by classic co-precipitation and alternating magnetic field assisted co-precipitation were very significant (Fig. 7).

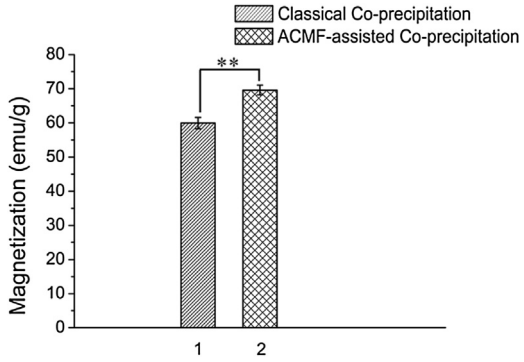


Fig. 7. Statistic data of magnetization of magnetic nanoparticle clusters prepared by classic co-precipitation(1) and alternating magnetic field assisted co-precipitation(2) ($p = 4.82*10^{-11} < 0.01$).

The magnetothermal effect of Fe_3O_4@PSC nanoparticle clusters prepared by ACMF assisted co-precipitation and classic co-precipitation were also characterized. Results showed that SAR of Fe_3O_4@PSC nanoparticle clusters prepared by alternating magnetic field assisted co-precipitation and classic co-precipitation were 402 and 345 W/g respectively (Fig. 8). The infrared thermal images indicated that the Fe_3O_4@PSC nanoparticle clusters prepared by alternating magnetic field assisted co-precipitation held potential as an agent of magnetic hyperthermia with good biocompability and high heating generation (Fig. 9).

Compared Fe_3O_4 nanoparticles clusters obtained by ACMF with ordinary product, we can easily get the conclusion that the for-

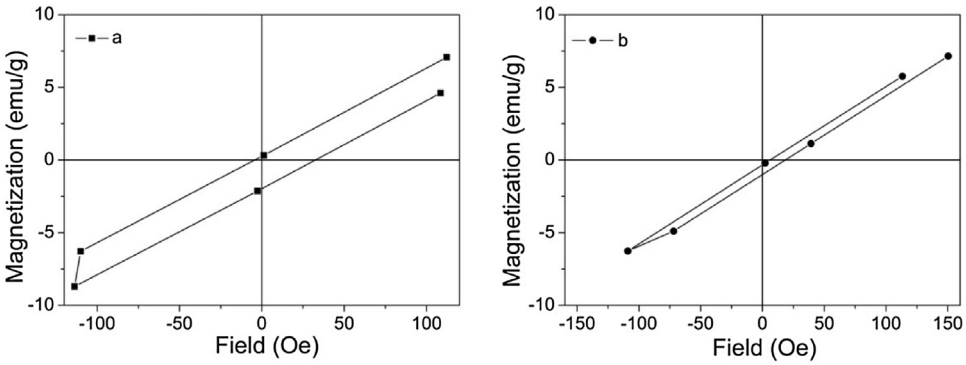


Fig. 6. Magnetic hysteresis loop of magnetic nanoparticle clusters prepared by alternating magnetic field assisted co-precipitation (a) and classic co-precipitation (b).

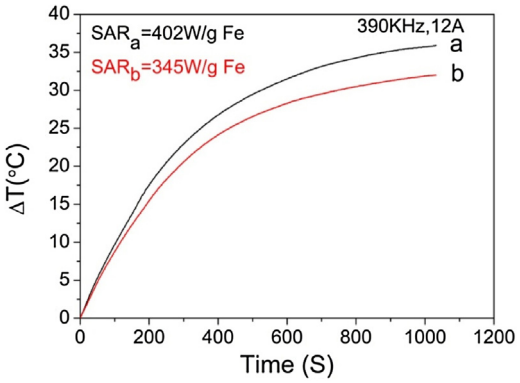


Fig. 8. Magnetothermal measurement of magnetic nanoparticle clusters prepared by alternating magnetic field assisted co-precipitation (a) and classic co-precipitation (b) under alternating magnetic field.

mer possess better magnetism. It is reported that FeOOH,the intermediate during the Fe_3O_4 preparation in co-precipitation would be magnetized by static magnetic field with low intensity (0–250 Oe),which would affect the growth of iron oxide nanoparticles, inducing the growth along the axis of being easily magnetized and further be attributed to the improvement of magnetic properties [26,27]. And the similar phenomena occurred in the ACMF could be explained well according to this theory.

In view of the above speculations, we could deduce the possible process and mechanism,which was shown in Scheme 1. Initially, iron ion solution was added into alkali solutions at room temperature and a short burst of nucleation occurred followed Lamer mechanism. Moderate radio-frequency device started and ACMF was produced. After fast nucleation, the tiny iron oxide nuclear could generate heat through relaxation loss adhered to Brown and Néel relaxation theory to make the solution temperature rise, meanwhile these smaller magnetized Fe_3O_4 nuclear could grow slowly along the axis of being easily magnetized of biggish nanoparticles depending on the heat produced by themselves, during this procedure nuclear fused into bigger nanoparticle with the Ostwald Ripening. When the aging process ended, these particles grew to the size of approximately 10 nm and presented uniform size distribution confirmed by TEM Characterization (Fig. 1a). Lastly, PSC was mixed for coating on the surface of particles. PSC plays an important role in hinder effect and makes the particles stable existence in solution without bigger aggregates. After the coating step finished, iron oxide nanoparticle clusters of 12 nm in single iron core size formed because of high surface energy belongs to every nanoparticle and could be seen in SEM Characterization (Fig. 2a). These nanoparticle clusters demonstrated higher saturation magnetization intensity compared to ordinary Fe_3O_4 nanoparticles prepared in oil bath, it may be concluded that Fe_3O_4 nanoparticles tend to grow along the magnetization direction induced by ACMF.

4. Conclusions

In conclusion, novel Fe_3O_4@PSC nanoparticle clusters were prepared in ACMF by co-precipitation. The magnetic properties and magnetothermol effect of Fe_3O_4@PSC nanoparticle clusters was studied and compared with Fe_3O_4@PSC nanoparticle clusters prepared by classic co-precipitation of similar size. As a result, Fe_3O_4@PSC nanoparticle clusters prepared in ACMF by co-precipitation displayed better saturation magnetization, coercive force, SAR and heating production under ACMF. As an explanation, the improvement in magnetic properties of samples prepared in ACMF might be due to the influence of ACMF on magnetic particle during the aging time, making nuclear tend to grow along magnetization direction to form nanoparticle with high magnetic

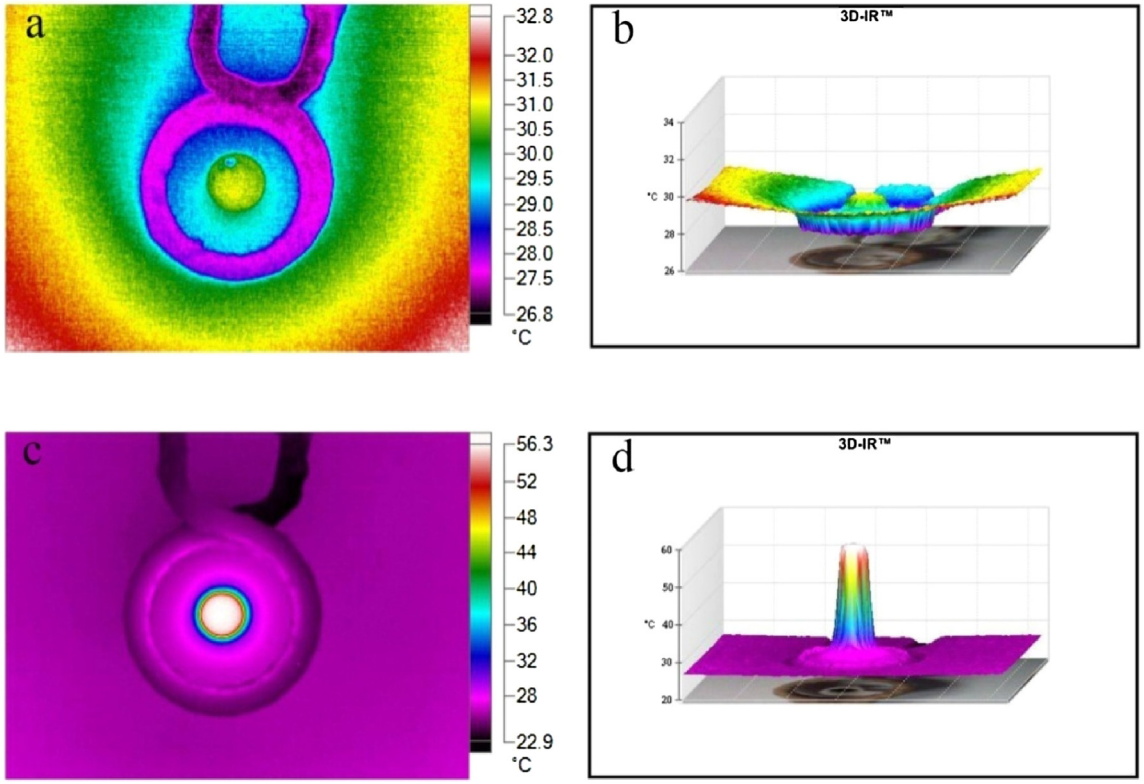
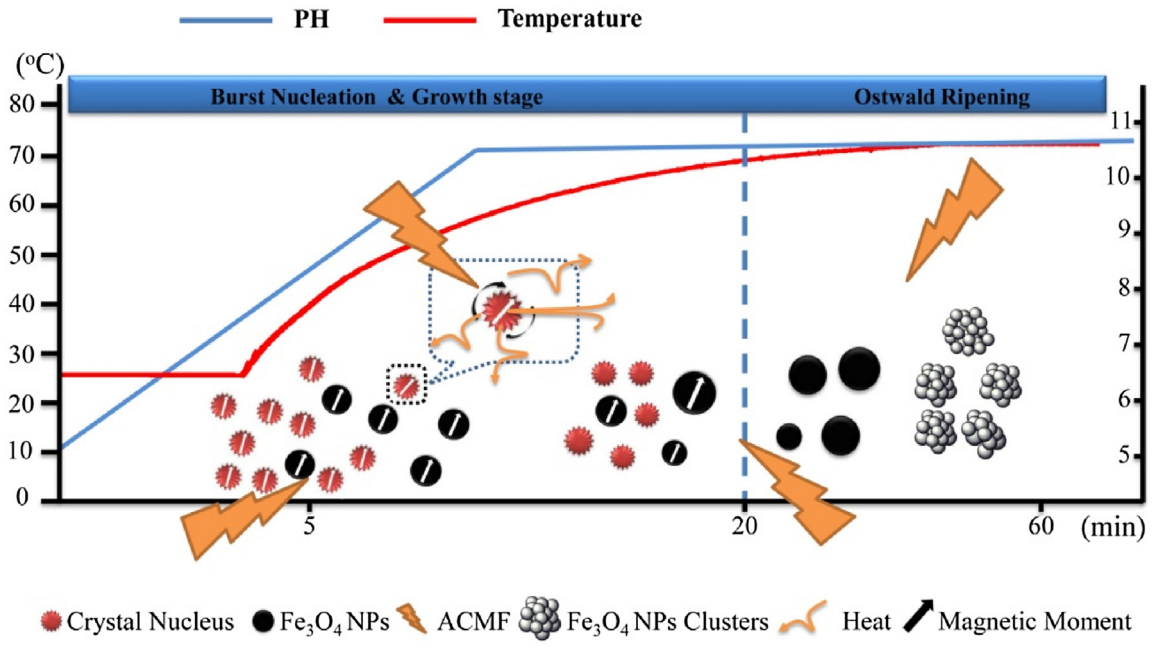


Fig. 9. Two dimensional and three dimensional infrared thermal images of magnetic nanoparticle clusters prepared by alternating magnetic field assisted co-precipitation before (a, b) and after applying ACMF for 10 min (c, d).



Scheme 1. Schematic illustration of preparation Fe_3O_4 nanoparticle clusters induced by ACMF. The data include temperature controlled by an ACMF generator (SPG-10-II) and pH adjusted by NH_4OH aqueous solution.

properties. This strategy based on ACMF might hold promise in preparation high performance magnetic nanomaterial and for more extensive application.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.colsurfa.2017.01.073.

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