Synthesis of 1-D Magnetic Nanoparticles for Torque Generation

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감사의 글

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항상 연구에 있어서 기초를 강조하시면서 학생의 입장에서 최적의 환경을 제공하여 주시고, 많이 부족한 저에게 도움과 가르침을 주신 교수님께 감사의 말씀 전합니다. 교수님의 지도아래 연구자로서 학문적인 발전뿐만 아니라 정신적으로도 한 단계 성장할 수 있었습니다. 교수님께서 항상 강조하시던 마음가짐을 잊지 않고 어디서든 최선을 다하는 제가 되도록 하겠습니다.

바쁘신 와중에 졸업 논문 심사를 맡아주신 유동원, 김형범 교수님께도 감사의 말씀 전합니다. 교수님들의 조언을 바탕으로 더욱 더 심도 있게 제 연구를 발전시킬 수 있었습니다.

항상 랩장으로써 굳은 일은 도맡아 하는 지욱이형, 피자 좋아하는 미현 누나, 근성과 햄버거의 태현이형, 고목나무의 매미 수진선배, 산유국 혜민선배, 매드

클라운 호영이, 술라프 슬미, 배불뚝이 정욱이, 안경잡이 희경이, 그리고 누나 집착남 대현이까지 바이오팀 멤버들 모두 감사하고 남은 학위기간 동안 좋은 연구와 좋은 일 가득하길 바랍니다.

인자한 미소의 승호이형, 근육이 남다른 승현이형, 잘생긴 용준이형, 미친 에너지를 가진 조증의 지효까지 자성팀을 포함해서, 층상팀의 에이스 재효형, 살찐 보거스 민지선배, 척척학사 봉관, 테니스 재원까지 모두 너무 감사드리고 덕분에 2년동안 잘 지낼 수 있었습니다. 선배들 뿐만 아니라 후배들도 어떤 어려움이 닥쳐도 함께 이겨나가며 학위 기간 동안 많은 것을 이루리라 믿습니다.

그리고 석사 기간 동안 진심으로 저에게 조언과 도움을 주신 유동원 교수님께도 감사드립니다. 항상 건강하시고 좋은 연구 하시기를 바랍니다.

그리고 항상 저를 뒤에서 지지해 주시고 기도해 주시는 아빠, 엄마. 어렸을 때부터 오랫동안 떨어져 살았지만 언제나처럼 저를 믿고 지켜봐 주시고, 제가 지치고 힘들 때마다 옆에서 응원해 주셔서 감사합니다. 많이 부족하고 배울게 많지만 매사에 최선을 다하고 아빠 엄마께 더욱 자랑스러운 아들이 되기 위해 열심히 사는 아들이 되겠습니다. 그리고 고민이 있을 때 마다 옆에 있어준 용방, 항상 긍정 에너지를 주는 추노, 그리고 영원한 보살 베네형까지 모두 함께해 줘서 고맙습니다.

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2016년 12월 27일

학위논문을 마무리하며

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Abstract

Synthesis of 1-D Magnetic Nanoparticles for Torque Generation

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Fabrication of 1-dimensional (1D) magnetic iron oxide nanoparticles is still a challenging and an important research subject in nanoscience as they have unique size and shape dependent characteristics. To address above issue, people have been synthesized iron oxide nanorods using solvothermal method or ionic liquid as a solvent. In addition, this issue can also be explored if we utilize an anisotropic unit cell structure that has an asymmetric shape instead of isotropic structure. In this report, we represent a method for synthesizing rod shaped magnetic iron oxide nanoparticles by fabrication of

FeO/α-Fe_2O_3 nanorods and phase transition of as-synthesized nanoparticles. In addition, specific surfactant and experimental environment was chosen to promote anisotropic growth of nanocrystals. Synthesized nanorods exert strong magnetic torque force which would be practically utilized to study mechanical manipulation of biological systems.

Keywords: 1D material, iron oxide nanoparticle, colloidal synthesis, torque force study, shape determinant of nanoparticles, rotating magnetic field

Chapter 1. Introduction

Magnetic nanoparticles have drawn lots of research interest in biological field and have been widely explored in various area.[1,2] New opportunities using magnetic nanoparticle are emerged because they can be manipulated under the external magnetic field. The location and activation of magnetic nanoparticles can be regulated by focusing the external magnetic field while magnetic field does not interfered by surrounding environment and has relatively deep penetration depth into biological organisms[3,4]. Utilizing these unique characteristics of magnetic nanoparticles allow for application of nanoparticles in the biomedical fields including magnetic resonance imaging (MRI)[5,6], heat emission in magnetic hyperthermia[7-9], and magnetically driven drug delivery system[10,11], etc.

Application of different types of magnetic force draw considerable attention to application of iron oxide magnetic nanoparticles using their magnetic force. Gradient force generated by magnetic probe successfully demonstrated direct observation of audio frequency tonotopy of chick hair cell.[12] In addition, interparticle force triggers magnetic switch and apoptosis signal under homogeneous magnetic field.[13]

Developing magnetic torque force has claimed particular attention to study mechanical manipulation of biological material, from proteins to double stranded DNA.[14] Altough magnetic tweezers (MTW) are traditionally used to study biological systems, this method can be further improved if higher torque force ranges around ~10 pN and dimension mismatch of micron-sized magnetic beads are solved[15]. Also, magnetic nanoparticles with high aspect ratio is an ideal morphology of nanoparticle for torque force related study according to traditional magnetic torque equation. Even though there are previously reported 1-dimensional magnetic nanoparticles[16,17], 0-dimensional magnetic nanoparticles are usually achieved due to its isotropic unit cell structure. A different strategy is necessary to fabricate magnetite nanorods.

In this study, we demonstrate an experimental method to fabricate 1-dimensional magnetic iron oxide nanoparticles. This method involves the synthesis of amorphous $FeO/\alpha\text{-}Fe_2O_3$ crystal structure via colloidal synthetic method utilizing metal precursor hot injection, followed by mild oxidation to produce Fe_3O_4 crystal structure[18]. This Fe_3O_4 nanorod not only has magnetization value, but also provides significant force to manipulate a single molecule event under homogeneous rotating magnetic field.

Chapter 2. Experimental and Equipment

2.1. Reagent

Iron pentacarbonyl (99.99%), oleic acid (90%), oleylamine (70%, tech. grade), 1,2-hexadecanediol (90%, tech. grade), trioctylamine (98%) are purchased by Sigma Aldrich.

2.2 Synthesis of 1-dimensional magnetic iron oxide nanorod

1-dimensional FeO/α-Fe_2O_3 crystalline nanorod is synthesized at first. Synthesis is processed using three-neck round-bottom flask under Ar flow with Schlenk line, inhibiting inflowing of oxygen and moisture. Metal precursor $Fe(CO)_5$ is hot injected and oleic acid, oleyl amine, and 1,2-hexadecanediol are used as surfactants while trioctylamine as solvent. There surfactants act as capping ligands that control the growth rate and determine the shape of nanoparticles. By adjusting the reaction time and amount of surfactant, iron oxide nanoparticles grow in anisotropic shape.

As-synthesized FeO/α-Fe_2O_3 nanorod is stirred at 120°C for 2 hours for oxidation of nanoparticles to obtain Fe_3O_4 crystalline nanostructures.

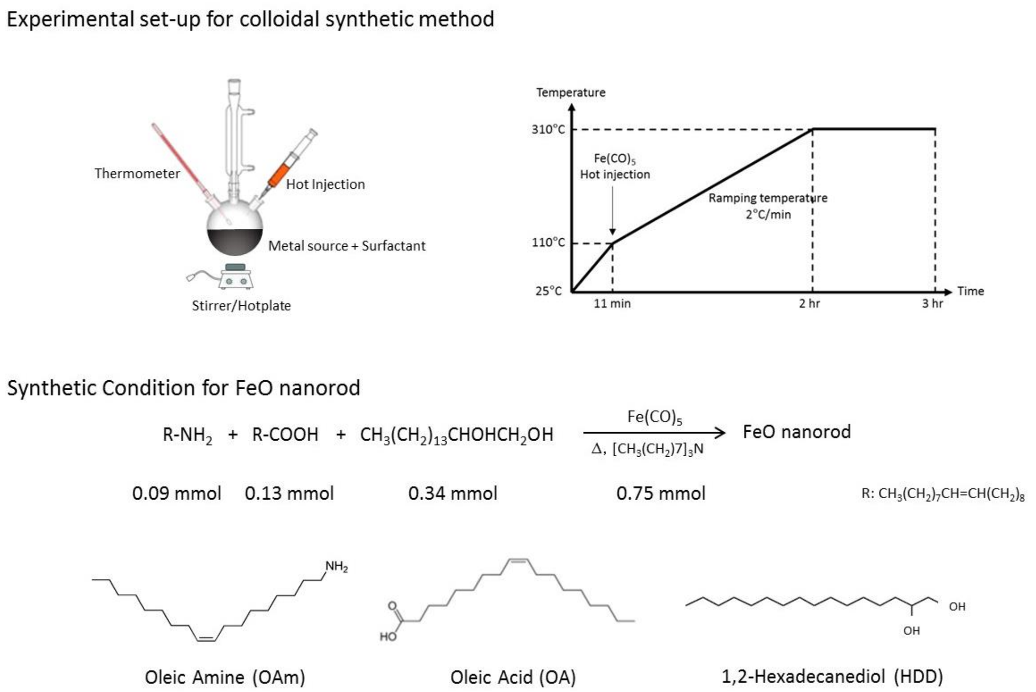
2.2.1 Synthesis of FeO/α-Fe_2O_3 crystalline nanorod

The mixture for the synthesis of FeO/α-Fe_2O_3 crystalline nanorod contain 8.3mL of trioctylamine, 0.13 mmol of oleic acid, 0.09 mmol of oleylamine and 0.34mmol of 1,2-

hexadecanediol in a three-neck round-bottom flask with Ar purging to remove the impurities like air and the other things. The reaction mixture was heated to 310°C in 2 hour after injection of 0.75 mmol $Fe(CO)_5$ at 110 °C and kept at this temperature for another 1 hour before the reaction is terminated. After the reaction products are cooled to room temperature, the black product is precipitated and isolated by centrifugation several times with ethanol. The isolated nanoparticles are dispersed in a solvent such as toluene with small amount of oleic acid and oleylamine to achieve better colloidal stability.

2.2.2 Oxidation of FeO/α-Fe_2O_3 nanorod to obtain Fe_3O_4 nanorod

Fe_3O_4 nanorod is obtained by oxidation of as-synthesized nanoparticles. After the reaction solution is exchanged from toluene to 1-octadecene, it was heated to 120 °C in air, and allowed to react for 2 hours.



Scheme 1. Synthesis of 1-dimensional iron oxide nanoparticles.

2.3. Analysis and Instruments

2.3.1 Analysis of shape and structure

1-dimensional magnetic iron oxide nanoparticles are analyzed by transmission electron microscopy (TEM), high resolution transmission electron microscopy (HRTEM) by using a JEM 2100 at 200 kV and a JEOL-ARMI 1300S at 1250 kV. X-ray diffraction (XRD) and vibrating sample magnetometer (VSM) are analyzed with Rigaku 2005G303 (Cu-*k*, 30kV, 15mA) and Lake Shore 7400-S Series.

For TEM and HRTEM, after aggregated iron oxide nanorods are removed by centrifugation at 50 rcf for 1 min, the supernatant is then re-centrifuged at 200 rcf for 5min. Mixture of sample with toluene is dropped onto Cu-ultrathin grid and Si-wafer and dried. For XRD and VSM data, the product solution is dried under vacuum for 1 hour and the dried sample is collected.

Chapter 3. Results and Discussion

3.1. Synthesis of rod shaped magnetic iron oxide nanoparticle

3.1.1 Strategy to synthesize rod shaped Fe_3O_4 nanoparticles

It has been difficult to synthesize 1-dimensional magnetic nanoparticles because magnetite (Fe_3O_4) has an inverse spinel group and face-centered cubic unit cell. Due to its intrinsic isotropic shape of the unit cell structure, usually 0-dimensional nanostructures are made. Among the other types of crystal structures of iron oxide, wustite (FeO) has a defected rhombohedral, anisotropic unit cell structure which allows progressive elongation along c-axis (Figure 1a). For example, ZnS nanocrystal, which has similar unit cell structure with wustite, has different surface energy[19]. This results in significant growth rate difference between different crystallographic directions. This experimental growth condition can be maximized using oleylamine (Figure 1b)[20]. In addition, α-Fe_2O_3 nanorod and Fe_3O_4 nanoprism are synthesized using surface selective surfactant such as oleic acid and 1,2-hexadecanediol (Figure 1c)[21]. These surfactants are known to have preferential binding affinity to {100} and {110} plane of crystals, which can also be found at wustite structures[22]. Although antiferromagnetic FeO crystal structure does not have magnetization value, it can be oxidized to Fe_3O_4 state, which is known to have saturation magnetization value among iron oxide crystal structure.

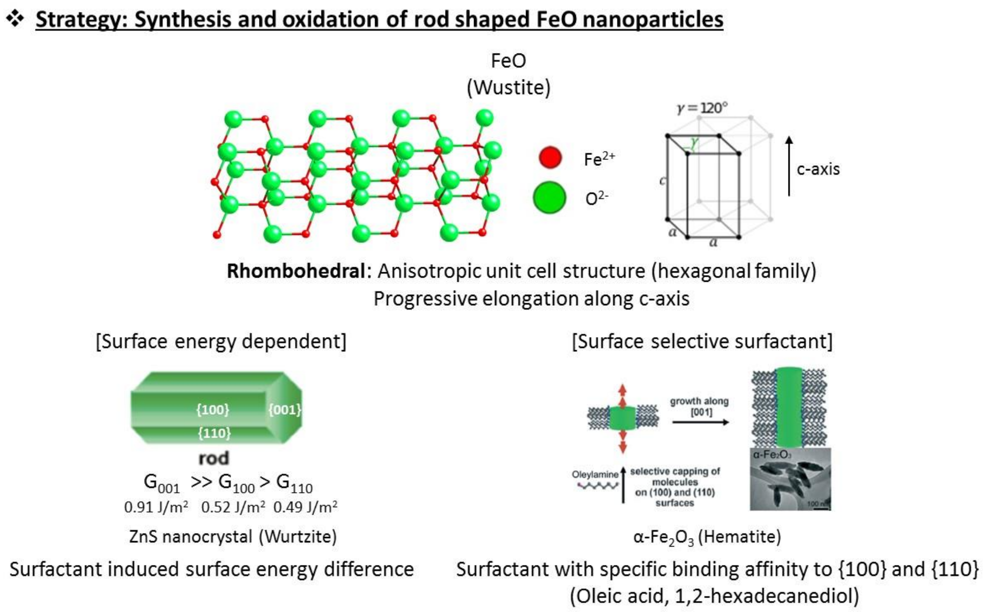


Figure 1. Strategy to synthesize rod shaped Fe_3O_4 nanoparticle.

3.1.2 Synthesis of rod shaped FeO/α-Fe_2O_3 nanoparticles

1-dimensional rod shaped amorphous FeO/α-Fe_2O_3 nanoparticles is conducted using $Fe(CO)_5$ as a metal precursor. Color changes of the reaction solution are the indication of the particle formation. After the hot injection of $Fe(CO)_5$ at 110°C, the transparent solution containing trioctylamine, oleic acid, oleylamine, and 1,2-hexadecanediol turn into yellow. Brownish color is observed when the reaction temperature reached around 180°C and turn into black very quickly after 200°C. The iron oxide nanorods are synthesized using 6:2.7:1:0.7 molar ratio of $Fe(CO)_5$:1,2-hexadecanediol:oleic acid:oleylamine. According to TEM analysis, these nanorods have an average diameter of 10±1 nm and three different aspect ratios (1:8, 1:11, 1:24) (Figure 1). HR-TEM study indicates that the nanorods have amorphous structure (Figure 2a). The lattice spacing for the core crystalline planes shown in Figure 2b are 2.145 and 1.517Å which can be assigned to {100} and {110} plane of FeO[23]. On the other hand, the outer shell's lattice spacing shown in Figure 1c are 2.51 and 2.54Å which can be assigned to {120} and {110} plane of hexagonal α-Fe_2O_3, respectively[23].

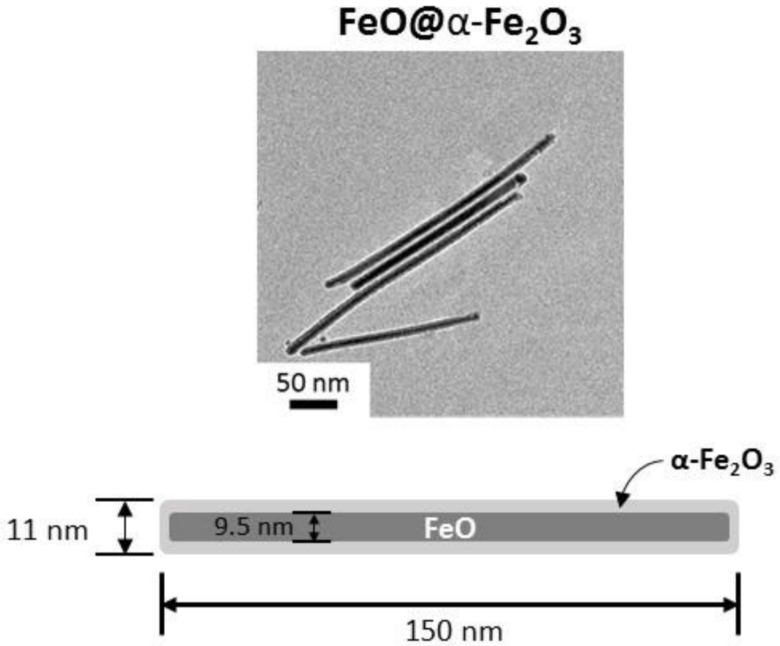


Figure 2. TEM image of rod shaped FeO/α-Fe_2O_3 nanoparticles. Average diameter and long axis is 11 nm and 150 nm, respectively. Core of nanoparticle has FeO crystal structure while outer shell has α-Fe_2O_3.

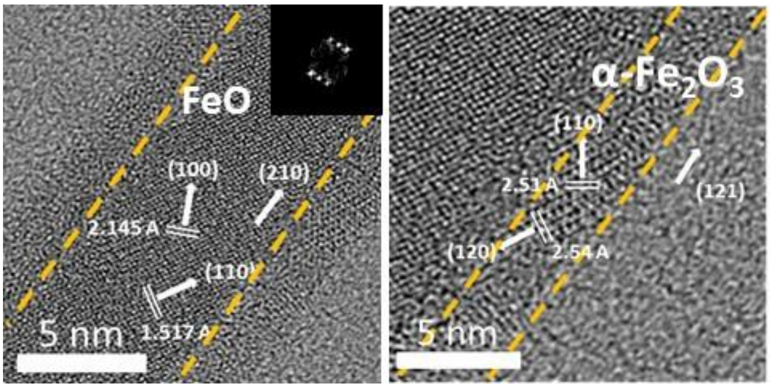


Figure 3. HRTEM image and fast Fourier transform (FFT) pattern of rod shaped FeO/α-Fe_2O_3 nanoparticles.

3.1.3 Synthesis of rod shaped Fe_3O_4 nanoparticles

The as-synthesized FeO/α-Fe_2O_3 nanorods are oxidized to obtain Fe_3O_4 nanorods. After the solvent containing the as-synthesized nanorods is switched from toluene to 1-octadecene, it is gently heated to 120°C in air and allowed to react for 2 hours. There is any color change from the reaction solution while heating. After the reaction is over, oxidation state of Fe was confirmed by observing solution attracted by a permanent magnet. According to TEM analysis, dimension of magnetic iron oxide nanorods are not changed (Figure 4). HR-TEM study indicates that the nanorods have crystalline (Figure 5). The lattice spacing for the core crystalline planes shown in Figure 5 are 2.51 and 1.75Å which can be assigned to {311} and {422} plane of cubic Fe_3O_4[23]. On the other hand, the outer shell's still have hexagonal α-Fe_2O_3, same as as-synthesized nanorods. The XRD patterns of each FeO/α-Fe_2O_3 and Fe_3O_4 are labeled with respect to standard ICDD cards (Fig 6 a,b)[22]. Especially, phase change to Fe_3O_4 are supported by five crystal planes of {220}, {311}, {400}, {511}, and {440} (Figure 6b)[23]. In addition, hysteresis curve of Fe_3O_4 nanorods at room temperature are shown in Figure 7. The saturation magnetization is 55 emu/g at 15000 Oe and coercivity indicates a ferromagnetic behavior of nanoparticles at room temperature.

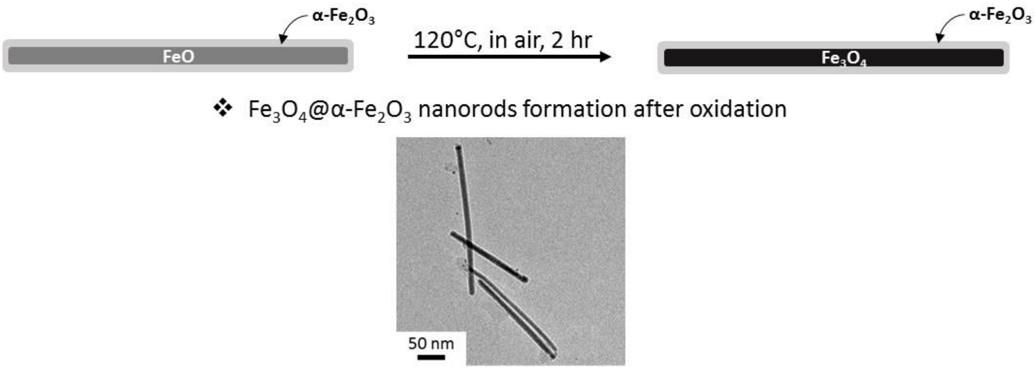


Figure 4. TEM image of rod shaped Fe_3O_4 nanoparticles. It was oxidized from as-synthesized nanoparticles. Core of nanoparticle has Fe_3O_4 crystal structure while outer shell has still α-Fe_2O_3.

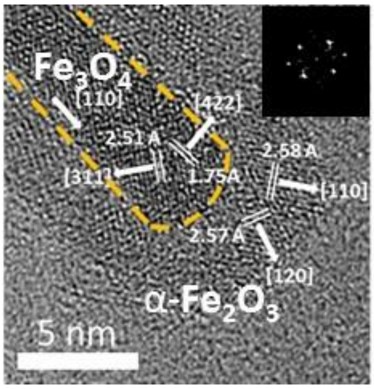


Figure 5. Structural analysis of rod shaped Fe_3O_4 nanoparticles. HRTEM image and fast Fourier transform (FFT) pattern of the nanoparticles.

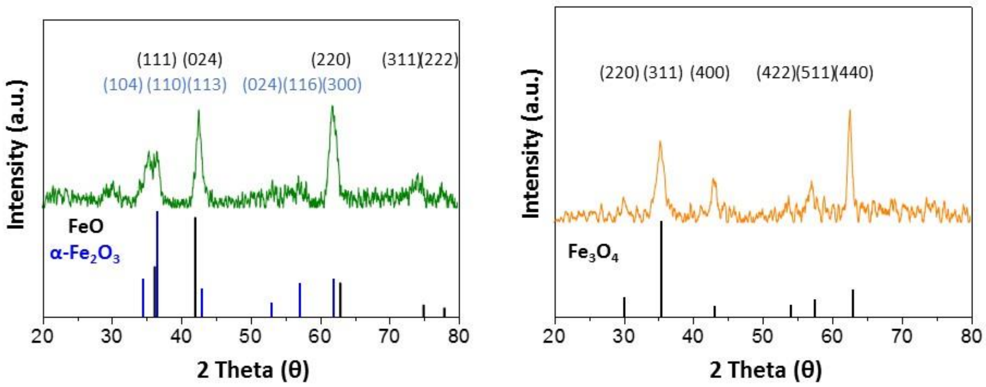


Figure 6. XRD data of the iron oxide nanorods. (a) FeO/α-Fe_2O_3 nanorod (b) Fe_3O_4 nanorod.

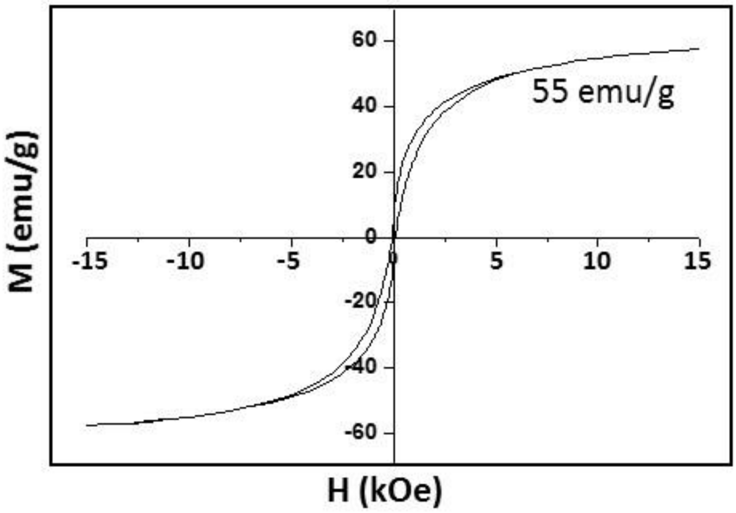


Figure 7. VSM data of the magnetite iron oxide nanoparticles.

3.2. Shape determinant of rod shaped iron oxide nanoparticle

3.2.1 Evolution of rod shaped iron oxide nanoparticle

To understand the formation of the iron oxide nanorods, we study the reaction systems at the early stages of the reactions. According to the TEM images in Fig 8, the very first set of tiny clusters formed at around 15 minutes after the reaction temperature reach at 310°C. These tiny clusters started to grow as the reaction time increases. Rod shaped nanoparticles start to emerge after 30 minutes. It seems that the nanorods are formed through the cluster of the tiny particles. On the other hand, we are only able to observe very small seeds below 310°C.

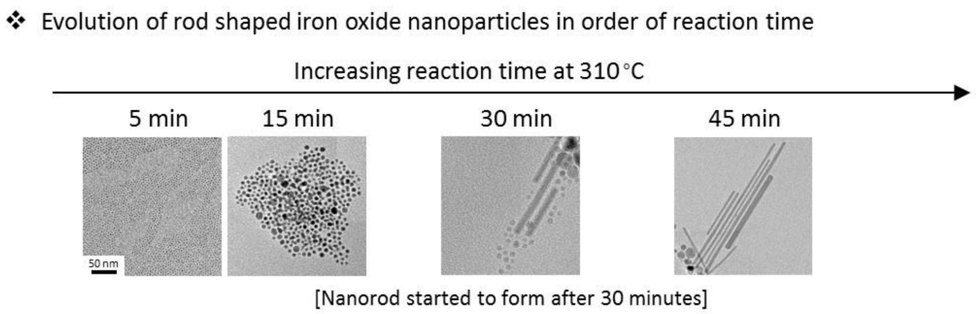
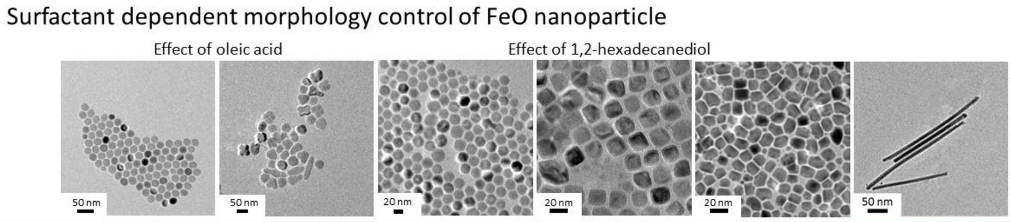


Figure 8. Evolution of rod shaped iron oxide nanoparticle**.** Rod shaped started to form after 30 minutes at 310°C. scale bar: 50 nm

3.2.2 Shape of iron oxide nanoparticles depends on the amount of surfactants

We also study the surfactant dependent morphology control of the nanoparticles. We used 6:2:1:0.7 molar ratio of $Fe(CO)_5$:1,2-hexadecanediol:oleic acid:oleylamine to synthesize the 1-dimensional iron oxide nanoparticles. We believe the concentration of metal source should be higher than the surfactant to let them to selectively bind to specific facets of particles which allows the anisotropic growth. To observe the nanoparticles' shape alteration, we change the concentration of oleic amine and 1,2-hexadecanediol while concentration of $Fe(CO)_5$ is fixed. While low surfactant concentration is used, 1-dimensional iron oxide nanoparticles are observed as the dominant growth are proceeded along the most reactive facets. On the other hand, when high surfactant concentration are used, 0-dimensional iron oxide nanoparticles are observed because excess amount of surfactant non-specifically bind to all facets that would make the particle to have similar growth rate (Fig 9).



OA	2.6	1.3	0.13			0.13
HDD	0.34		1.4	1	0.7	0.34

Oleic acid (OA) 1,2-Hexadecanediol (HDD) Unit: mmol

Figure 9. Shape of iron oxide nanoparticles depends on the amount of surfactants.

3.3. Torque force calculation of magnetite iron oxide nanoparticles and magnet set-up to generate rotating magnetic field

We calculate the torque force exerted by rod shaped magnetic iron oxide nanoparticles. After oxidation of the nanoparticles, they are centrifuged with different speed for size selection. Aspect ratio of 1:24 and 1:8 were obtained using 300 rcf and 100 rcf for 2 minutes while dominant number of nanoparticles with aspect ratio 1:11 are gained using 200 rcf for 2 minutes. Magnetic torque force equation is $\boldsymbol{\tau} = \mathbf{V} * \mathbf{M} * \mathbf{B}$, when V indicates volume of particle, M means magnetization of body, and B applied magnetic field. After calculation, we obtain that magnetic torque force with aspect ratio of 1:8, 1:11, 1:24 had torque 98.4, 42.4, and 48.2 pN·nm, respectively (Fig. 10)[24]. These torque force exerted by nanorods are very promising compared to previously reported nanorods, as they can generate higher torque and can be practically used for various kinds of biological applications. In addition, we built three different types of set-up of magnet system to generate rotating magnetic field (Fig. 11). 3 axis electromagnet has millimeter range of working distance between sample and coils while it generates 2mT homogeneous magnetic field (Fig 11a). Rotation of permanent magnet can observe diffraction patterns of nanoparticle utilizing specific laser source (Fig 11b). Although its working range is limited, 2 axis electromagnet probe can generate strong 300mT homogeneous magnetic field using aligned electromagnet probe tips (Fig 11c).

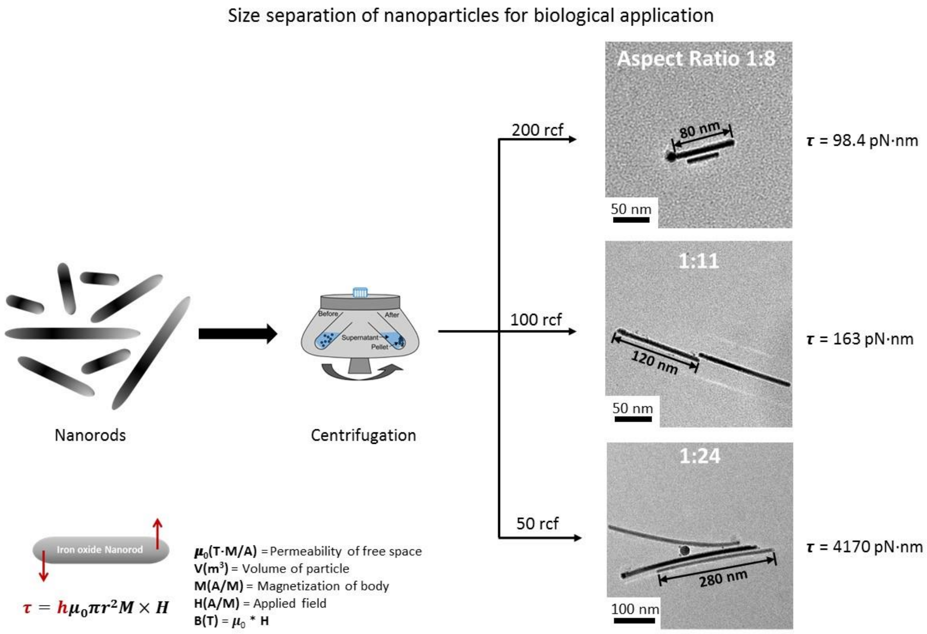


Figure 10. Magnetic torque force variation using different aspect ratio

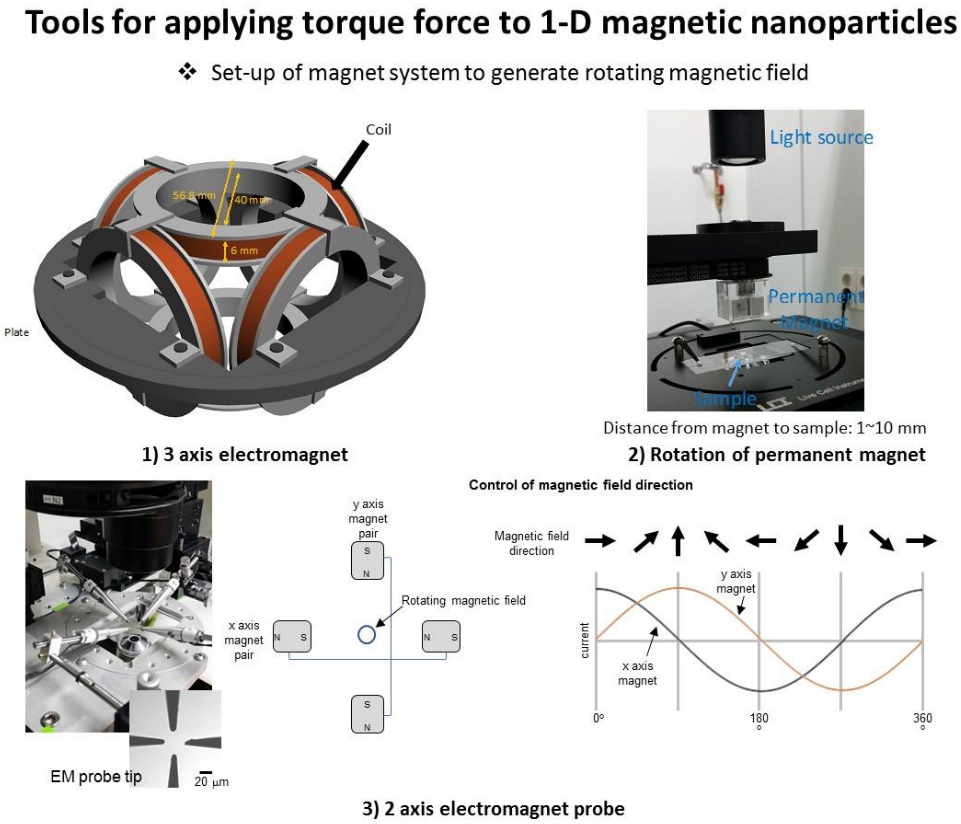


Figure 11. Tools for applying torque force to 1-dimensional magnetic iron oxide nanoparticle

Chpater 4. Conclusion

We report the method for producing rod shaped magnetic iron oxide nanoparticle, which yields 11 nm diameter and 1:11 aspect ratio. Synthesized FeO/α-Fe_2O_3 nanorods using their rhombohedral anisotropic unit cell structure and oxidation of as-synthesized nanoparticles are conducted to produce Fe_3O_4 nanorods. Characteristics of nanorods are confirmed through TEM, HRTEM, XRD, and VSM. To understand the particle formation mechanism, we monitor the evolution of nanorods at 310℃ while high amount of surfactant is used to observe whether 0-dimensional nanoparticles are formed. Finally, we estimate magnetic torque force of the nanorods, which had average 42.4 pN and it is strong enough for application of various biological area.

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국문 요약

회전력 응용이 가능한 1차원 구조의 자성나노입자 합성

최홍서

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크기와 모양에 따라 특이한 성질을 가지는 1차원 구조의 자성나노입자의 합성은 여전히 도전적이며 동시에 나노 분야에서 중요한 연구 주제이다. 1차원 구조의 자성나노입자를 합성하는데 어려운 이유는 산화철의 단위 격자 구조가 등방성인 이유로 보통 영 차원 적인 구조가 여러 조건에서 쉽게 합성이 되기 때문이다.

따라서, 본 연구에서는 비대칭 구조를 가지게 하여 주는 $FeO/\alpha\text{-}Fe_2O_3$ 조성의 나노입자 합성 후에 산화 과정을 통하여 큰 자성을 띄는 1차원 구조의 자성나노입자 합성을 보여준다. 이를 위하여, 특정한 종류와 농도의 계면 활성제를 이용하여 나노입자의 비대칭 구조를 가지게 하여 주는 조건을 조성하였다. 합성된 1차원 구조의 자성나노입자는 자기장 아래 강한 회전력을 가지며, 이는 생물학적인 요소들의 다양한 역학을 연구할 수 있는 도구가 될 수 있을 것이다.

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핵심되는 말: 일차원 물질, 콜로이달 합성, 회전력 연구, 나노입자의 모양을 결정짓는 요소, 회전 자기장