Research Article

# Synthesis and characterization of hydrothermally synthesized α-$Fe_2O_3$ nanostructures with controlled morphology

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**Abstract**
Antimicrobial α-$Fe_2O_3$ nanoparticles were synthesized using hydrothermal method (HS) at 150 °C for 12 h. $FeCl_3$ and urea were used to have α-$Fe_2O_3$ nanostructures with the help of appropriate amount of surfactants to control the morphology. Stable colloidal suspensions were prepared using synthesized nanoparticles and then 3-D metallic filters were coated using electrophoretic deposition under definite voltage and time. It was shown that the obtained particles were effective to eliminate *S. Aureus Bacterias*.

**Keywords** α-$Fe_2O_3$ · Nanoparticles · Coating · EPD · Antibacterial

## 1 Introduction

α-$Fe_2O_3$ nanoparticles have become critical materials in recent years for various applications and therefore it is widely used due to its unique characteristics, such as high corrosion resistant and antimicrobial behavior [1, 2]. α-$Fe_2O_3$ nanoparticles are also used as a photocatalyst n-type semiconductor because its bandgap (nearly 2.1 eV) permits considerable amount of the solar energy [3, 4]. It also shows desirable photocatalytic efficiency and stability in aqueous solutions [4, 5]. Synthesis method and the type of surfactant material used during synthesis play highly important role in order to have oriented morphology with desired properties. For instance, Pu et al. [2] synthesized pure α-$Fe_2O_3$ nanoparticles with different morphology using different amount of CTAB as a surfactant material. Spray and Choi [4] used anodic deposition method to synthesize highly transparent and uniform films composed of α-$Fe_2O_3$ particles.

In the present work, α-$Fe_2O_3$ nanoparticles were obtained at 150 °C for 12 h using HS in an attempt to control the morphology using TWEEN80 as a surfactant. It is also shown that α-$Fe_2O_3$ nanoparticles are effective to eliminate *S. Aureus Bacterias*.

## 2 Experimental section

α-$Fe_2O_3$ nanoparticles were synthesized hydrothermally at 150 °C for 12 h. $FeCl_3$ and urea were used as starting precursors where both deionized water and butanol were solvents. $FeCl_3$ and urea were solved into the mixture of deionized water and butanol. TWEEN 80 was added as a surfactant material. The obtained solution was put into a Teflon-lined autoclave and sealed. Hydrothermal synthesis has taken place at 150 °C for 12 h. After the synthesis, the color of the obtained solution was changed into red from yellow whilst the pH of the solution was also change to 8.5 from acidic pH range. The solution was washed with distilled water and the red precipitate was dried at 70 °C for 24 h. Dried nanoparticles were calcined at 350 °C for 1 h.

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Using hydrothermally synthesized α-$Fe_2O_3$ nanoparticles stable colloidal suspensions (0.5 wt%) were prepared where the 2-Proponal used as a solvent. TWEEN80 was used as a surfactant material once again in order to prevent the flocculation. The prepared stable colloidal suspensions were used for the electrophoretic deposition (EPD) process. 3-D nickel based filter materials were coated using EPD using different coating times from 3 to 5 min with a fixed voltage of 30 V DC.

## 3 Results and discussion

The photos of the obtained solutions before HS, after HS and the final product after drying are shown in Fig. 1a–c, respectively, clearly indicating that change in the color is taking place proofing that the transition is actually occurred.

Figure 2 indicates both SEM and TEM micrographs of the synthesized α-$Fe_2O_3$ nanoparticles. Figure 2a reveals that, α-$Fe_2O_3$ nanoparticles were clearly synthesized in near spherical morphology while the average particle size is determined to be about 90 nm, as shown in Fig. 2b. However, some degree of aggregation caused by the calcination process is also seen in Fig. 2a, b. In order to understand the shape and size of the nanoparticles in detail TEM observations are also conducted as shown in Figs. 2c, d which indicate that the shape of α-$Fe_2O_3$ nanoparticles have spherical morphology with an average particle size of 90 nm. However, there are also some particles with bigger diameter as high as 150 nm, as shown in Fig. 2d.

The crystallinity, phase and the purity of the sample was determined using XRD analysis (Fig. 3). XRD patterns exhibited that intense peaks belong to the α-$Fe_2O_3$ nanoparticles according to the standards (JCPDS card no of α-$Fe_2O_3$: 33-0664). Only one peak at around 2θ = 56° could be attributed to the γ- $Fe_2O_3$ phase, as marked in graphic (JCPDS card no of γ-$Fe_2O_3$: 39-1346). No other characteristic peaks were observed for impurities.

Figure 4 indicates the antimicrobial activity of hydrothermally synthesized and calcined α-$Fe_2O_3$ nanoparticles. It can be seen that as the concentration of nanoparticles were increased, number of bacteria colonies were decreased. The most efficient concentration was seen in 1000 µg/ml of α-$Fe_2O_3$. That concentration was eliminated almost more than half of the control colony.

Figure 5a shows the photo of nickel based metallic filter coated using EPD under 30 V for 3 min. Figure 5b shows secondary electron image of the coated filter whereas Fig. 5c indicates back-scattered image of the filter. Macro image of the coated filter (Fig. 5a) shows that the coating process was occurred successfully. SE and BSE micrographs of the filter's also proof that the success of the EPD. As seen in Fig. 5c the inner parts of the filter coated, too.

## 4 Conclusion

α-$Fe_2O_3$ nanoparticles with an average particle size of 90 nm were synthesized at 150 °C for 12 h by hydrothermal treatment. TWEEN80 was used as a surfactant material in order to have spherical nanoparticles. The antimicrobial effect was determined against to *S. Aureus Bacterias* and it was shown that α-$Fe_2O_3$ nanoparticles have remarkable antimicrobial efficiency in high concentrations. Stable colloidal suspensions were obtained whereas 2-propanol used as a suspension media and Ni-based filters were coated using an applied voltage of 30 V by EPD.

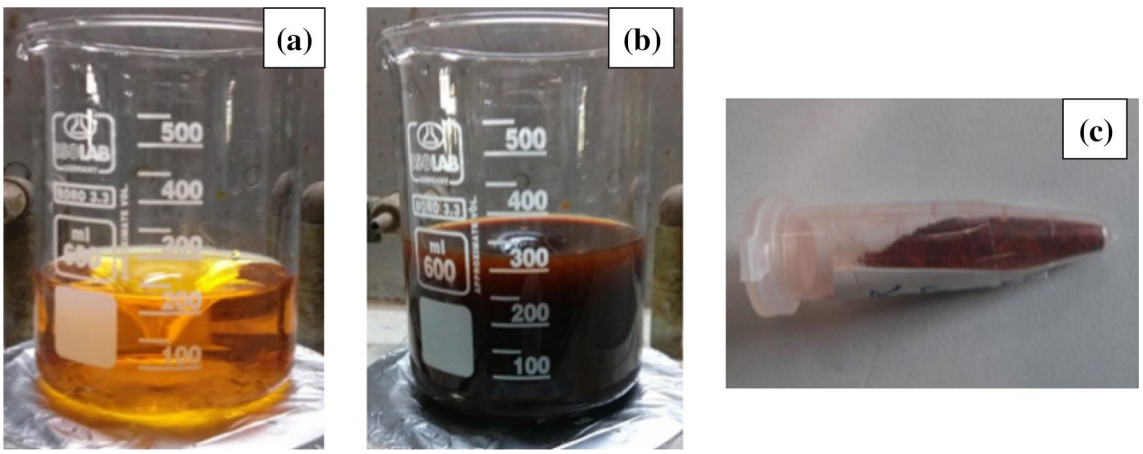


**Fig. 1** The photos of the solution before (**a**), after (**b**) the hydrothermal synthesis and the final dried nanoparticles (**c**)

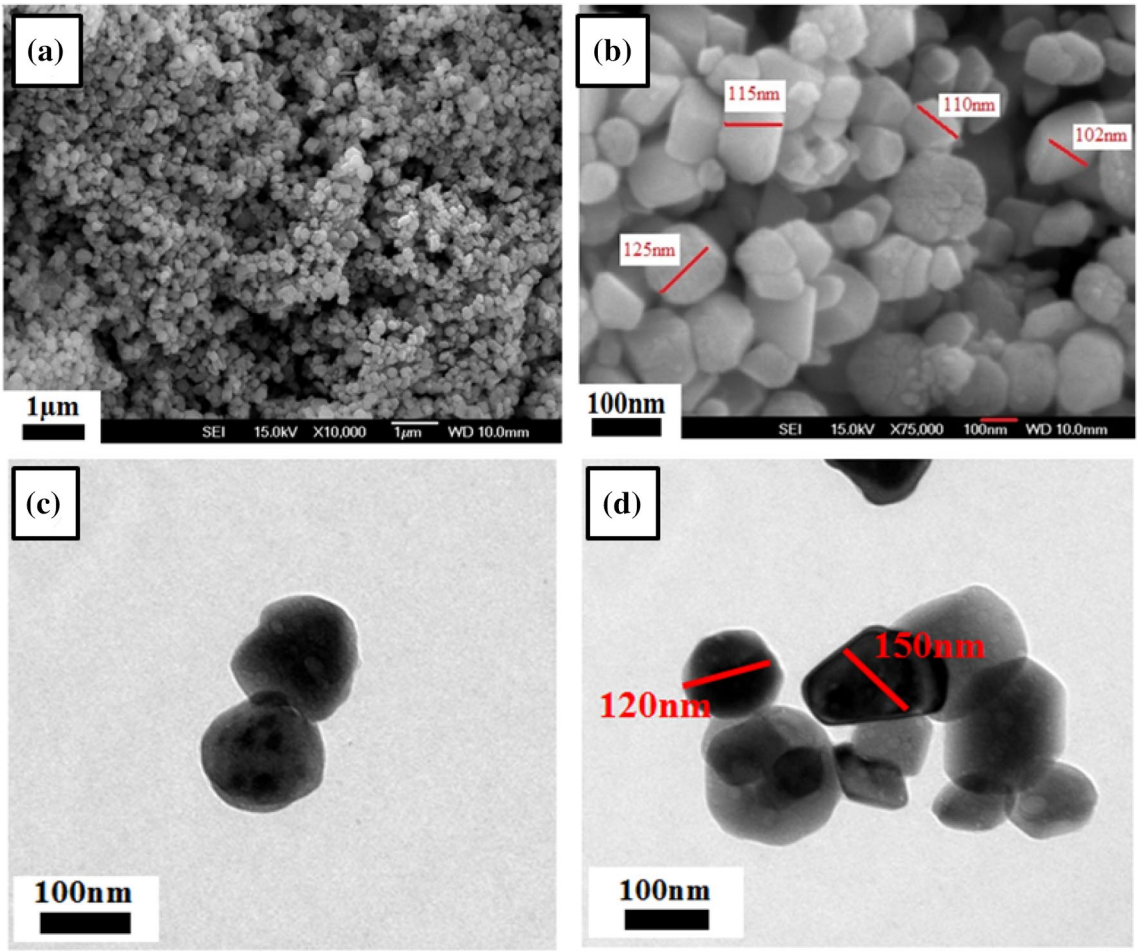


**Fig. 2** SEM micrographs (**a**, **b**) and TEM micrographs (**c**, **d**) of hydrothermally synthesized α-$Fe_2O_3$ nanoparticles

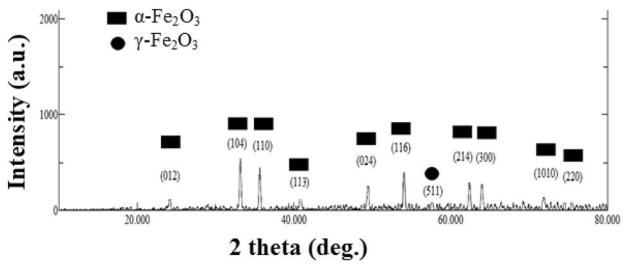


**Fig. 3** XRD patterns of α-$Fe_2O_3$ nanoparticles

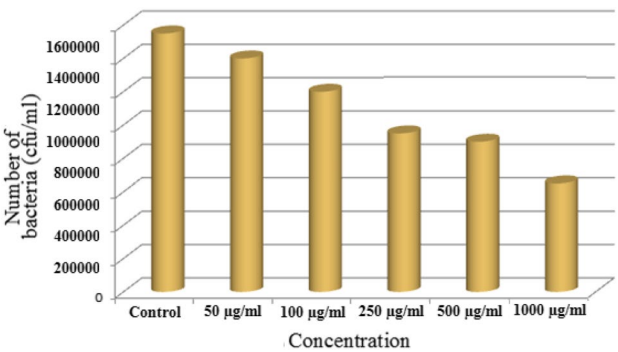


**Fig. 4** Antimicrobial effect of calcined α-$Fe_2O_3$ nanoparticles against to *S. Aureus Bacterias*

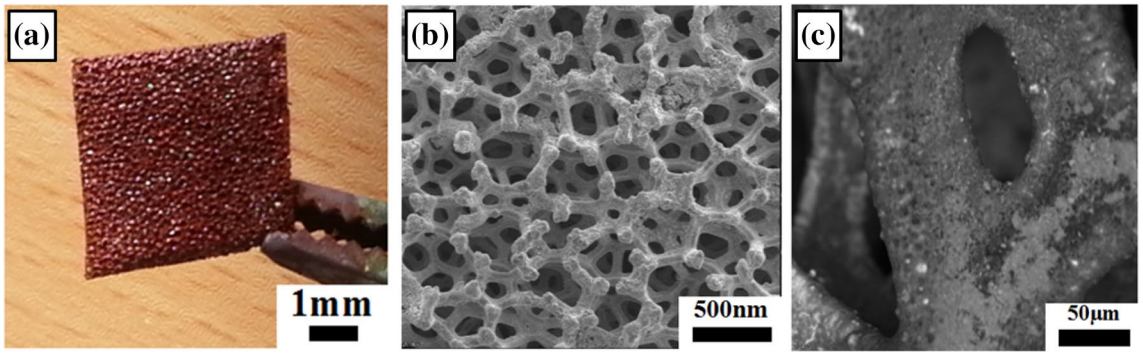


**Fig. 5** Images of the coated filter using EPD process; macro images (**a**), secondary electron (**b**) and back-scattered electron (**c**) images of the coated filters

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## Compliance with ethical standards

**Conflict of interest** The authors declare that they have no conflict of interest.

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