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## Synthesis of hematite-magnetite nanocomposite by low voltage dc arc method

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### ABSTRACT

The paper presents a new simple and cheap method to synthesize hematite-magnetite nanocomposite. Low voltage dc arc is exerted into the electrochemical cell between two iron tip electrodes in different solvents. The results of this works show that polar solvents such as water and dimethylformamide can be used as suitable media to establish a low voltage dc arc in small distance between two needle electrodes. In the polar solvents, lower dc voltage can make a low current arc between two electrodes with 2 mm distance. The synthesized iron oxide nanomaterials are in hydrated and amorphous form. The synthesized nanomaterial can converted into crystalline iron oxides. The morphology and the composition of the iron oxide samples are characterized by SEM and XRD.

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### KEYWORDS

Dc arc;  
Electrosynthesis;  
Hematite;  
Magnetite;  
Nanocomposite.

### INTRODUCTION

During 20 years ago, nanomaterials have received steadily growing interests in all science special in material science, physics, chemistry, environmental science, as well as in all engineering fields, due to their peculiar and fascinating properties, and potential applications. Among the nanomaterials, iron oxide nanoparticles exhibit unique characteristics, such as half-ferromagnetism, large specific area, nontoxicity and biocompatibility, and has a variety of potential applications, such as ferrofluids, catalysts, high-dense information storage, contrast agent, targeted drug delivery, solid phase extraction and other chemical and biomedical applications. Accordingly, preparation of iron oxide nanoparticles has become an

interest and active research field. Various techniques have been developed up till now, such as arc-discharge, chemical precipitation<sup>[1]</sup>, hydrothermal<sup>[2]</sup>, reverse microemulsion<sup>[3]</sup>, sol-gel<sup>[4]</sup>, electrochemical<sup>[5]</sup>, pulse electrosynthesis<sup>[6]</sup>, and etc. However, large-scale, low-cost and environmental friendly synthesis of iron oxide nanoparticles, especially with good size controlling and narrow size distribution, is still very difficult. Therefore, developing a new method to synthesize iron oxide nanoparticles is necessary and interest. Arc-discharge in water is a newly developed method for preparing carbon nanomaterials and nanooxides<sup>[7-9]</sup>. In the present work, a new dc arc method has been applied in different solvents to prepare hematite-magnetite nanocomposite.

## EXPERIMENTAL

All materials and reagents were purchased from Merk or Fluka. Double distilled water was used in all experiments. All the electrochemical experiments were carried out by an electrolyzer equipped with pulse system (BTE 04, Iran). A scanning electron microscope (SEM) from Philips (XL30, Netherlands) was used for the studying of morphology and particles sizes of the samples. X-ray diffraction (XRD) studies were performed by a Decker D<sub>8</sub> instrument.

## RESULTS AND DISCUSSION

When the arc-discharge method is used to synthesize nanomaterials, it has some factors which can affect on the morphology, particles sizes and the composition of the synthesized sample. The effective factors of the dc arc-discharge synthesis method include arc voltage, solvent, electrode material, the distance between anode and cathode and solvent temperature. When the arc is discharged between electrodes, the solution temperature is increased. The amount of flowed heat from discharged arc is depended on solvent dielectric, distance between electrodes and applied voltage. Therefore, the applied voltage and the electrode distance are the main factors. The following text describes the experimental procedures that used to investigate the effects of the important factors on the morphology and the composition of the samples. Figure 1 shows the experimental set up.



Figure 1 : Experimental set up.

Based on the initial studies, the distance between anode and cathode is an important factor as well as the applied voltage that can control the electrical current of the discharged arc. We checked different amounts of this factor from 0 up to 10 mm. At distances more than

2 mm, discharging process need a voltage higher than 100 V. We tried to establish a dc arc by using a voltage lower than 100 V. Therefore, we decreased the electrode distance from 5 mm to 1mm. By using 1mm distance between anode and cathode, dc arc can be established in polar solvents.

After optimizing the electrode distance, 80 V dc arc was exerted into the different solvents including acetone, acetonitrile, dimethyl sulfoxide (DMSO), tetrahydrofuran (THF), ethanol, methanol, dimethylformamide (DMF) and water. The amount of the established arc current was used as a controlling parameter to select the suitable solvent. No considerable discharge current was seen for methanol, ethanol, acetone, acetonitrile, DMSO and THF. The discharging arc in water and DMF showed acceptable currents (0.1 and 1 A for DMF and water, respectively). The synthesized samples were studied by SEM. Figure 2 shows the SEM images of the samples synthesized in

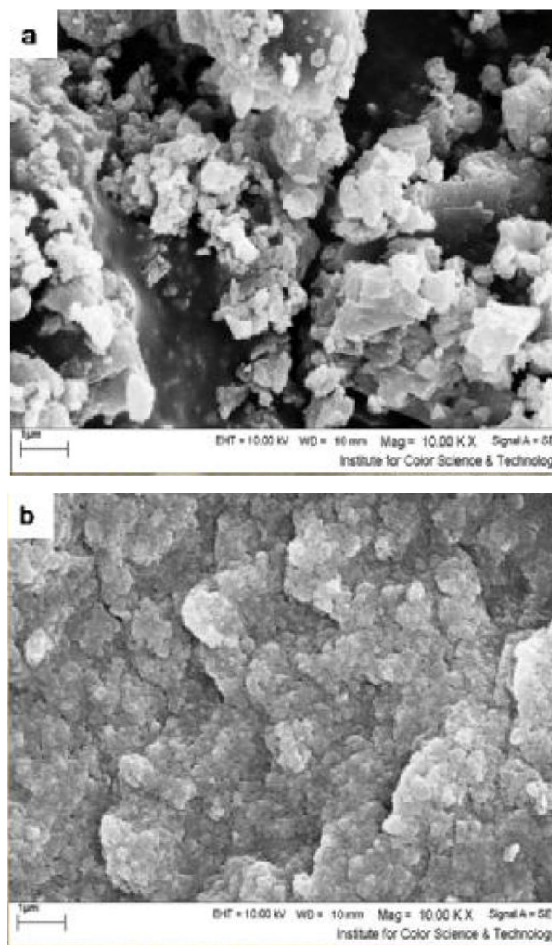


Figure 2 : SEM images of the iron oxide samples synthesized in DMF (a) and water (b).

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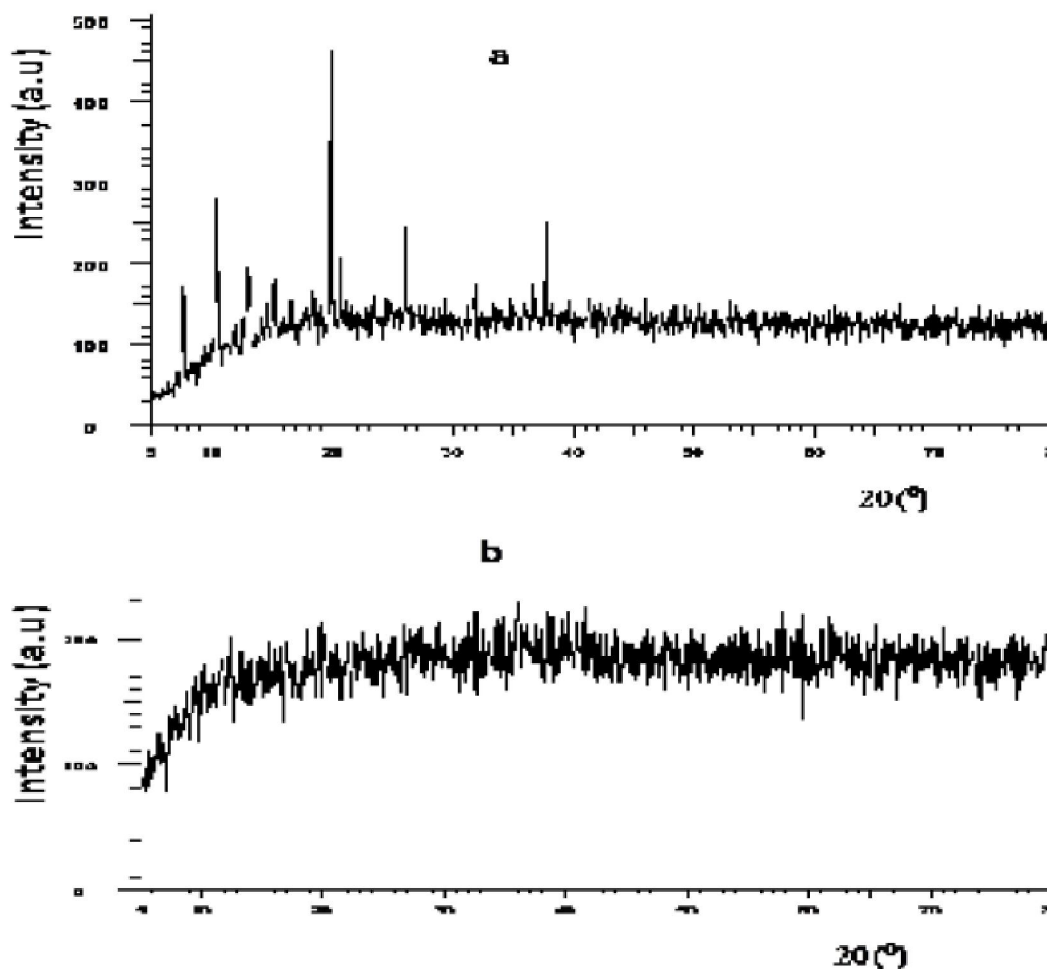


Figure 3 : XRD patterns for the synthesized iron oxide samples in DMF (a) and water (b).

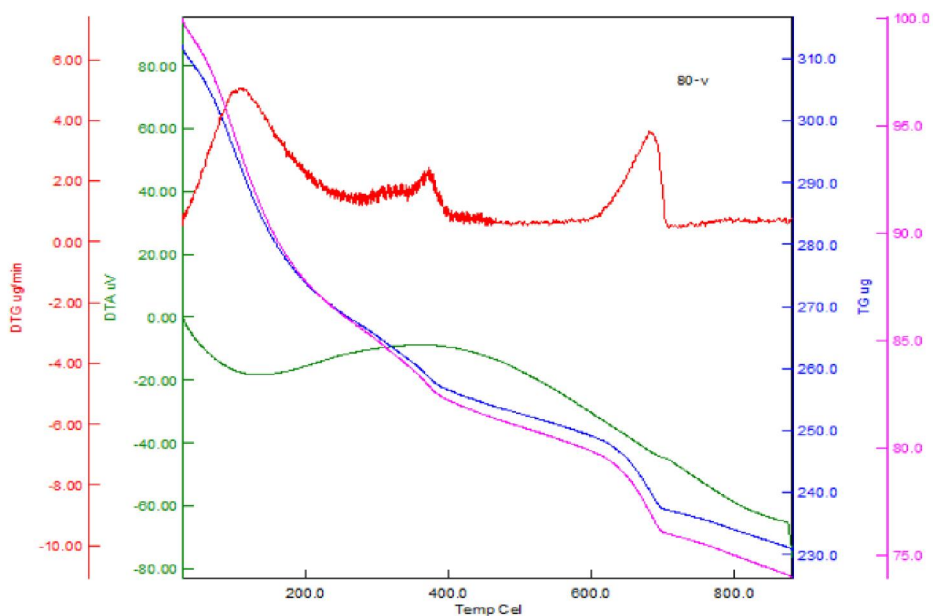


Figure 4 : TGA/DTA analysis of the iron oxide sample synthesized by arc discharge in water solvent.

water and DMF solvents.

As it can be seen in Figure 2, the sample synthesized

in water has uniform morphology and the smaller particles (30 nm average diameters). Therefore, the water is

more suitable solvent to synthesize iron oxide nanoparticles. The compositions of the samples were identified by XRD patterns (Figure 3). The XRD results showed that the synthesized sample in DMF has acceptable crystallinity, but the synthesized sample in water has low crystallinity. To obtain suitable temperature to increase sample crystallinity, the sample was analyzed by

TGA/DTA (Figure 4). Figure 4 shows that iron oxide in magnetite form are stable at 650 °C and lower. Therefore, to increase sample crystallinity, the sample was heated to 500 °C for 2 h. The heated sample was analyzed again by XRD. Figure 5 shows the XRD patterns of the sample. As it is obvious in Figure 5, the heated sample shows acceptable crystallinity. The sample in-

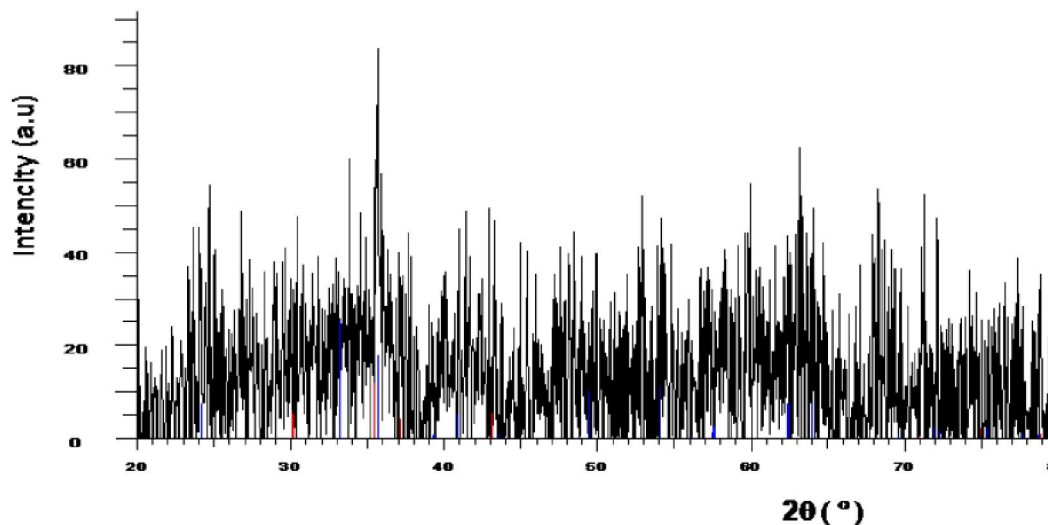


Figure 5 : XRD patterns of the iron oxide sample synthesized in water after heating in 500 °C for 2 h.

cludes 1:1 hematite and magnetite nanocomposite.

## CONCLUSIONS

Dc arc discharge in water can be used as a confident and controllable method to prepare hematite/magnetite nanocomposite. In this method, electrode distance, voltage and solvent type are effective parameters that can change the morphology and the particles sizes of the nanocomposite samples.

## ACKNOWLEDGEMENTS

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