# **Waste Water Treatment by Iron Nanoparticles Prepared by Pulsed Electrochemical Deposition**

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### **Abstract**

Recent industrial and urban activities have led to elevated concentrations of a wide range of contaminants in groundwater and wastewater, which affect the health of millions of people worldwide. In recent years, the use of zero-valent iron (ZVI) for the treatment of toxic contaminants in groundwater and wastewater has received wide attention and encouraging treatment efficiencies have been documented. In the present work, nanoscale iron was prepared by pulse electrodeposition of nano iron by chemical reduction of iron chloride and iron sulfate. Our research focuses on iron nanoparticles preparation and its use for wastewater treatment.

#### **Introduction**

Various technologies are currently available to remove arsenic from waste water, such as ion exchange [1], coagulation (coprecipitation) [2], reverse osmosis [3], bioremediation [4], and adsorption [6]. Adsorption is a common practice for arsenic removal from waste water due to technological and cost advantages. Because of their high affinity for arsenite (As(III)) and arsenate  $(As(V))$ , elemental iron and iron  $(hydr)$ oxides are widely applied as the adsorbents for arsenic removal [5], [6] and [7]. Nano iron powders have been used in permeable reactive barriers for intercepting arsenic plumes in contaminated groundwater [8] or added to household filters for arsenic removal in developing nations [9]. Nanoscale iron (NSI) was recently reported as an ideal candidate for *in situ* remediation of arsenic contaminated groundwater [10] and [11]. It can also be a promising material for arsenic removal from wastewater because of its large active surface area and high arsenic adsorption capacity.

All described properties of Fe (iron) nanoparticles can be even used for decomposition of pollutants contained in the waste water, mainly for treatment of industrial sewage and hutch water. The usage of nanoiron can represent a significant qualitative step in the classical technologies of water treatment including drinking water. NSI can be also used for a reduction of the content of heavy metals, nitrates and phosphates in the drinking water.

 Nanotechnology has widespread application potential and offers also the possibility of an efficient removal of pollutants and germs in the area of wastewater treatment. The objectives of this study were to prepare NSI by pulse electrodeposition and to test its performance for efficient removal of pollutants from the wastewater. Electrochemical deposition is a cost effective, competitive method, which allows control over composition and microstructure of the powder.

### **Experimental Synthesis of nano-iron powder**

Iron nanoparticles were produced by pulse electrodeposition. In pulse electrodeposition a D.C. current is applied for a short period time, t<sub>on</sub>, that is followed by a period of time when no current is applied,  $t_{off}$ . Typical values for  $t_{on}$ , and  $t_{off}$  are between 5 and 200 ms and between 1 and 10 ms respectively. Through the use of a high current density as well as some grain growth inhibitors, such as saccharine, it is possible to increase the nucleation rate and reduce grain growth.



Fig1. Pulse electrodeposition of nano iron. Fig. 2. Schematic figure of nano iron preparation.



$$
Fe(H2O)63+ + 3 BH4- + 3H2O \rightarrow Fe0 + 3B(OH)3 + 10.5 H2
$$
\n(1)\n
$$
Pd2+ + Fe0 \rightarrow Pd0 + Fe2+
$$
\n(2)

The structure of the materials prepared was investigated using X-ray powder diffraction using Co  $K_{\alpha}$  radiation and with a highresolution SEM from JEOLJSM-5600LV.

As mentioned, the bath composition for the Fe electrodeposition was composed mainly of:



Sacharin 0.3 g·dm<sup>-3</sup> and magnesium sulfate (MgSO<sub>4</sub>) betwee 0 and 0.5M was added as a grain refining agent. The temperature of the solution was  $25^{\circ}$ C and the pH varied from 3.5 to 6.0. Pulse current was between 10 and 20mA. All cases deposition were made on titanium cathode in order to facilitate their removal from the substrate which allows to perform experiments on self supported samples. One should also mention that the more additives are added to the solutions the larger is the contamination problem of the samples. For this reason, most of the experiments were done with the lowest amount of additive as possible.

#### **Results and discussion**

Pulse current electrodeposition of nano-Fe was carried out to investigate the effect of MgSO<sub>4</sub> on the microstructure and the reactivity of the electrodeposited films. Electrodeposition without in  $MgSO_4$  electrolyte gave smaller grain size than with it.

XRD results in Fig.3. demonstrated that no other component were present within the Fe1 and Fe2 samples. We used  $MgSO_4$  as a grain refining agent. From the X-ray diffractorgram was calculated the grain size of the NSI using Debye-Scherrer equation:

$$
\tau = \frac{K \cdot \lambda_{Co}}{\beta \cdot \cos \theta} \tag{3}
$$

where,  $\tau$  is the grain size; K is a dimensionless shape factor, with a value 0.9,  $\lambda_{\text{Co}}$  is the X-ray wavelength (1.788897Å);  $\beta$  is the line broadening at half and  $\theta$  is the Bragg angle.

From the calculation turns out that the Fe1 grain size is about 11 nm and in the case of Fe2 samples it was 7-8 nm.



Fig.3. X-ray diffractogram of the NSI samples prepared without and with added MgSO<sub>4</sub>.



Fig.4. SEM results of the nano-scale Fe1 powder without added MgSO<sub>4</sub>.



Fig. 5. SEM results of the nano-scale Fe2 powder with added MgSO4.

The surface morphology of the nano-Fe was characterized with SEM (scanning electron microscope). The surface morphology changed as rough surface, where the concentration of MgSO4 increased. The reactivity of active surface of nano-Fe increasing as a function of decreasing of grain size in presence of MgSO4.

# **Conclusion**

There is an increasing interest in the use of NSI for the removal of contaminants from groundwater and wastewater. NSI has been successfully applied for the remediation/treatment of groundwater and wastewater contaminated with chlorinated organic compounds, nitroaromatic compounds, arsenic, heavy metals, nitrate, dyes, and phenol. The review shows that there is a need for more detailed systematic studies on contaminants removal mechanism and also some technical improvements in utilizing NSI.

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