

## Soil Remediation Using Nano Zero-valent Iron Synthesized by an Ultrasonic Method

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### Abstract

A new method for the synthesis of nano zero-valent iron (nZVI) was developed in the present study. Ultrasonic waves, as a novel method, were used to synthesize the nanoparticles. The morphology and surface compositions of the particles were characterized by using FESEM, XRD, BET, and particle size analyzer. The synthesized nanoparticles were then utilized as a Fenton-like catalyst to degrade hydrocarbon contaminants of soil. The effect of using ultrasonic waves in combination with nZVI for hydrocarbon degradation was also investigated. The effects of ultrasonic power, nZVI concentration, pH, hydrogen peroxide concentration, and temperature on remediation were studied. It was found that the new nZVI synthesized by an ultrasound-assisted method had high efficiency in soil remediation. The results indicated that the efficiency of removing hydrocarbons by nZVI was 98.57%. Finally, the optimum conditions of degradation were obtained when pH, ultrasonic power, nZVI concentration, temperature, and hydrogen peroxide concentration were respectively equal to 3.5, 500 W, 0.4 gr.l<sup>-1</sup>, 40 °C, and 30 mM.

**Keywords:** Synthesis, Nanotechnology, Hydrocarbon Contaminants, Degradation, Ultrasonic, Soil Remediation, nZVI, Fenton

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### 1. Introduction

Soil contamination threats to human health through food and groundwater are one of the most important environmental concerns. Hydrocarbons are present in soil and may usually be found in urban and rural lands due to the leakage of gasoline, motor oil, and diesel fuel from storage tanks (Chang, et al. 2007). Most of the common methods developed for treating the soil contaminated with hydrocarbons have some restrictions (Ellen, et al. 1995). For example, using landfill cannot remove all contaminants (Fairbanks, et al. 1971); the incineration of the pollutants may produce secondary pollutants such as poly-aromatics (Ellen, et al. 1995). In biological treatment, due to the resistance of some pollutants against microorganisms and the reduction of the activity of microorganisms in different climatic conditions, the efficiency of removing pollutants may be low (Lim, et al. 2005); in the thermal methods of removing volatile contaminants, high operating temperatures are required, which is not economic (Fairbanks, et al. 1971). A practical and modern alternative method to remove hydrocarbons from contaminated soil is using nanotechnology-based methods. Nano zero-valent iron particles have been used in treatment technologies (Fairbanks, et al. 1971; Chang, et al. 2007). Various studies show that nanoparticles are very effective in the removal of environmental pollutants such as

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chlorinated solvents such as poly chlorinated biphenyl (PCB) and trichloroethane (TCE), inorganic compound such as perchlorate, and heavy metals like chromium, lead, nitrate, copper, and zinc Ellen, et al. 1995, (Liao, et al. 2007). The effectiveness of nZVI particles may be related to their small sizes and large surface area to volume ratios. nZVI particles can be easily synthesized using sodium borohydride and NaBH<sub>4</sub> as strong reducing agents (Lim, et al. 2005). In order to produce nZVI with a specific surface area and morphology, various chemical and physical methods are used in the literature (Masciangioli, et al. 2003). One of the characteristics of these particles is their tendency to aggregate and change to form a chain-like structure (Rasheed, et al. 2011; Wang, et al. 1997). This reduces stability and decreases interfacial area drastically (Liao, et al. 2007). The stability of nZVI particles against aggregation can be improved by imparting electrostatic repulsion using noble metals, polymers, ceramic materials, dextran, oils, and silicon dioxide (Chang, et al. 2007; Rasheed, et al. 2011). Another promising method that serves this goal is to synthesize nZVI particles in the presence of supporting inorganic materials (Masciangioli, et al. 2003; Schrick, et al. 2002).

The main aim of this work is to investigate the performance of the degradation of hydrocarbon contaminants in soil by using the nZVI synthesized by employing ultrasonic wave. The effects of ultrasonic power, nZVI, H<sub>2</sub>O<sub>2</sub> concentration, and temperature on the efficiency of the degradation are also investigated.

## 2. Material and methods

### 2.1. Chemicals and equipment

FeSO<sub>4</sub>.7H<sub>2</sub>O (MW: 278.01, Analar), ethanol (absolute, Merck), H<sub>2</sub>O<sub>2</sub> (30%, Merck), sodium hydroxide (0.1 M, Merck), and hydrochloric acid (36% wt/wt, Merck) were used as received without further purification. All the required solutions were prepared using deionized and deoxygenated water. In order to generate ultrasonic waves in solution, the UIP1000hd sonication bath of Hielschler Company was used.

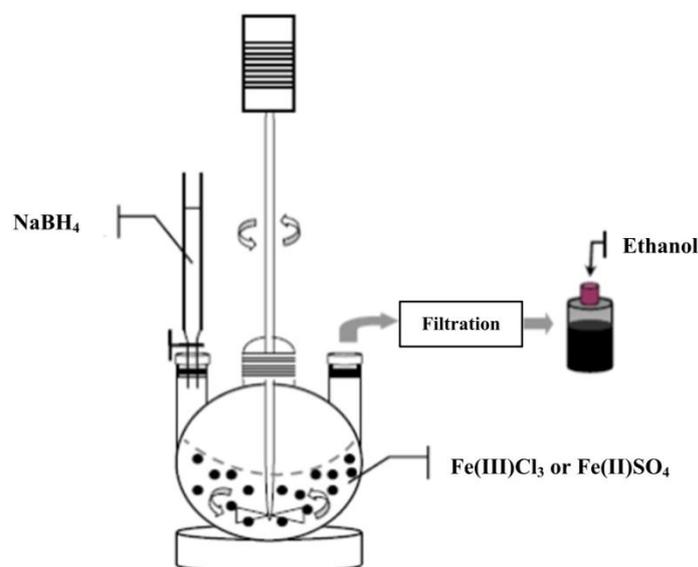
### 2.2. Method of nZVI preparation

The first step in the synthesis of the nanoparticles is to prepare the solutions of FeSO<sub>4</sub> and NaBH<sub>4</sub>. A solution of 2.25 gr. FeSO<sub>4</sub> (0.08M) in 100 ml of ethanol (30%) was prepared and mixed by an agitator for 5 minutes to be homogeneous enough. For the preparation of NaBH<sub>4</sub> solution, 1.5 gr. of NaBH<sub>4</sub> (0.4M) was solved in 100 ml deionized water which had already been deoxygenated by using nitrogen gas for 30 minutes.

nZVI was synthesized in a 300-ml flask reactor with three open necks (Liao, et al. 2007). One of the necks was used for the ultrasonic probe; another one was employed to purge oxygen out by using nitrogen gas and to prevent the oxidation of the nanoparticles. The last neck was used for adding the NaBH<sub>4</sub> solution. A peristaltic pump was used to add 100 ml of the borohydride solution to 100 ml of the ferric ion solution. A schematic diagram of the experimental setup is shown in Figure 1. For adjusting the temperature at 25 °C, the reaction container was put in a water bath so that the solution temperature was kept constant during the reaction. By adding the NaBH<sub>4</sub> solution to the flask, black spots were immediately formed in the solution, which implied that the iron particles were deposited according to Equation 1.



At the end of the reaction, the slurry was put under ultrasonic waves for 5 minutes. The slurry was then filtered using a 0.2-micron membrane filter; the black cake was rinsed with ethanol (100%) for three times. The wet cake was centrifuged at 3000 rpm for 5 minutes to remove the remaining moisture and was then stored in a vacuum container to be dried. The dried cake was kept in a refrigerator to avoid oxidization (Fairbanks, et al. 1971). A small amount of the sample was used for analyses.

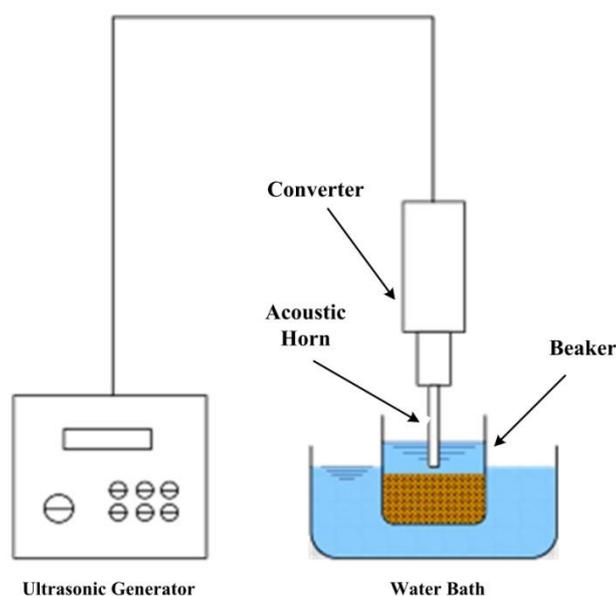


**Figure 1**

A schematic diagram of the experimental setup for the preparation of nZVI

### 2.3. Degradation experiments

In order to investigate the degradation efficiency of nZVI, 15 grams of dried contaminated soil that the physical and chemical properties of which are shown in Table 1, and 10 ml of crude oil that the properties of which are shown in Table 2, are used. The sample was added to a 200-ml glass beaker and was mixed (by a magnetic stirrer) with 70 ml of deionized water for 2 minutes. After complete mixing, pH reached 8 and it was then adjusted at 3 by adding hydrochloric acid. Next, the synthesized nZVI was introduced to the mixture. Herein, ultrasonic waves were also used to enhance the decontamination of soil. To this end, a sonic probe with a diameter of 0.5 inch was put in the reaction media as schematically shown in Figure 2. Due to the transfer of high ultrasonic power to the mixture, the temperature tended to increase; therefore, the reactor was placed in a water bath to carefully control the temperature at a constant set point (Fairbanks, et al. 1971; Hoffman et al. 2007). The mixture was exposed to ultrasonic power for 30 minutes; having removed the probe, hydrogen peroxide was then added to the solution. The resulting mixture was stirred by a magnetic stirrer for 30 minutes to have a total reaction time of 60 minutes. The mixture was subsequently filtered through a vacuum filter. In order to find the remained hydrocarbon in the remedied soil, it was washed several times by a 1:1 mixture of acetone and hexane. The solvent containing the extracted hydrocarbons was fed to a gas chromatograph. The extracted hydrocarbons were characterized by a TR-121033 gas chromatograph with a flame ionization detector (GC-FID). A TRB-5 capillary column with an ID of 30 m by 0.32 mm and a film thickness of 1  $\mu\text{m}$  was used for the separation of the hydrocarbons. Typically, 0.5  $\mu\text{l}$  of total petroleum hydrocarbon (TPH) extract was injected into the GC system (Rasheed, et al. 2011).

**Figure 2**

A schematic diagram of the experimental setup for the degradation experiments

**Table 1**

Soil physical and chemical properties

Sample	Texture			pH	Conductivity	Total Carbon (%)	Total Nitrogen (%)	
	Sand	Silt	Clay					
Clay	22.2	35.7	42.1	7.38	26.2	1.29	0.079	
Sample	Exchangeable Cations (mol/kg)					CEC (mol/kg)	ESP (mol/kg)	SAR (mol/kg)
	Al <sup>3+</sup>	Ca <sup>2+</sup>	Mg <sup>2+</sup>	K <sup>+</sup>	Na <sup>+</sup>			
Clay	0.025	15.43	2.552	0.379	0.166	18.55	0.9	3.9
Sample	Average Pore Size (Å)			Total Pore Volume (mm <sup>3</sup> /gr.)		Surface Area (m <sup>2</sup> /gr.)		
Clay	11.23			6.993		24.9		

## 2.4. nZVI characterization

After the preparation of nano zero-valent iron particles by using the ultrasonic method, their physico-chemical properties were investigated through particle size analyzing methods such as FESEM, XRD, and BET. An EQUINOX 3000 diffractometer (INEL France 2010) at 45 kV and 30 mA was used for the XRD analysis. The source consisted of Cu K $\alpha$  radiation ( $\lambda = 1.54 \text{ \AA}$ ) and the sample was scanned from  $0^\circ$  to  $118^\circ$  with a scanning rate of  $2^\circ$  per minute. The nZVI sample was thoroughly washed with ethanol before the XRD analysis to remove any impurities (Liao, et al. 2007). The FESEM analysis was carried out using a HITACHI 4160 field emission scanning electron microscope (FESEM). The particle size analysis was performed with a DT 1200 acoustic spectrometric (Dispersion Technology Inc.) which utilizes the sound pulses transmitted through a particle suspension to measure the properties of the suspended particles (Lim, et al. 2005). BET surface area ( $S_{\text{BET}}$ ) was measured by N<sub>2</sub> desorption method (Nova 2200, Quantachrome). Prior to the adsorption-desorption measurements, the samples were degassed by flowing N<sub>2</sub> through them for 3 hours at 200 °C.

**Table 2**  
Crude oil properties

Weight Percent Composition		Weight Percent Composition		
Element	Range	Hydrocarbon	Average	Range
Carbon	83% to 87%	Paraffins	30%	15% to 60%
Hydrogen	10% to 14%	Naphthenes	49%	30% to 60%
Nitrogen	0.1% to 2%	Aromatics	15%	3% to 30%
Oxygen	0.05% to 1.5%	Asphaltics	6%	Remainder
Sulfur	0.05% to 6.0%			
Metals	< 0.1%			

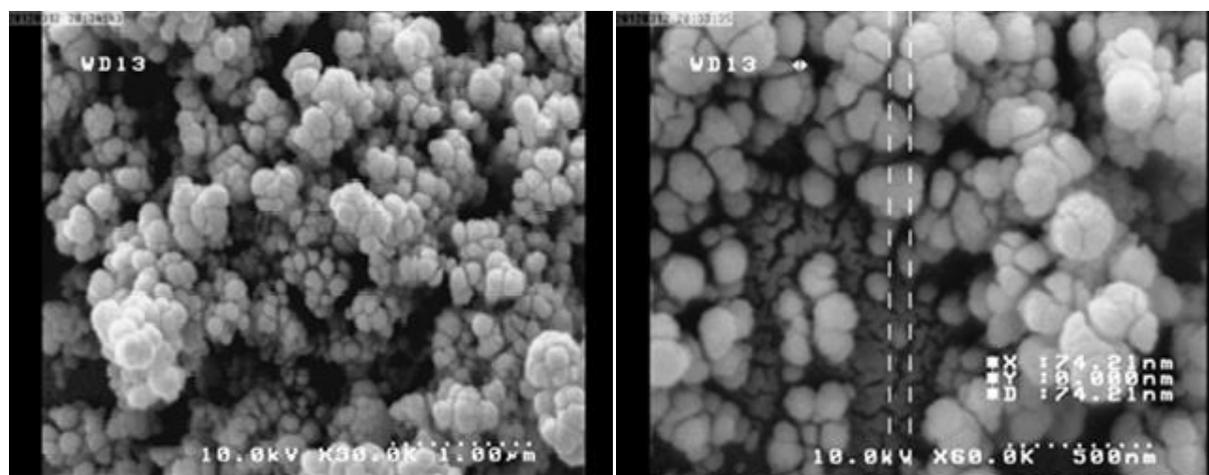
### 3. Results and discussion

#### 3.1. nZVI characterization

#### 3.2. nZVI morphology

The FESEM images of the nZVI particles at different magnifications are shown in Figure 3. It is clear that although the nZVI particles tend to form irregular clusters, they also demonstrate some dispersion; the particles range in size from 40 nm to 80 nm.

The specific surface area of the nanoparticles was determined with the classic BET method. The BET surface area of the green nanoparticles was  $35.8 \text{ m}^2 \cdot \text{gr}^{-1}$ .

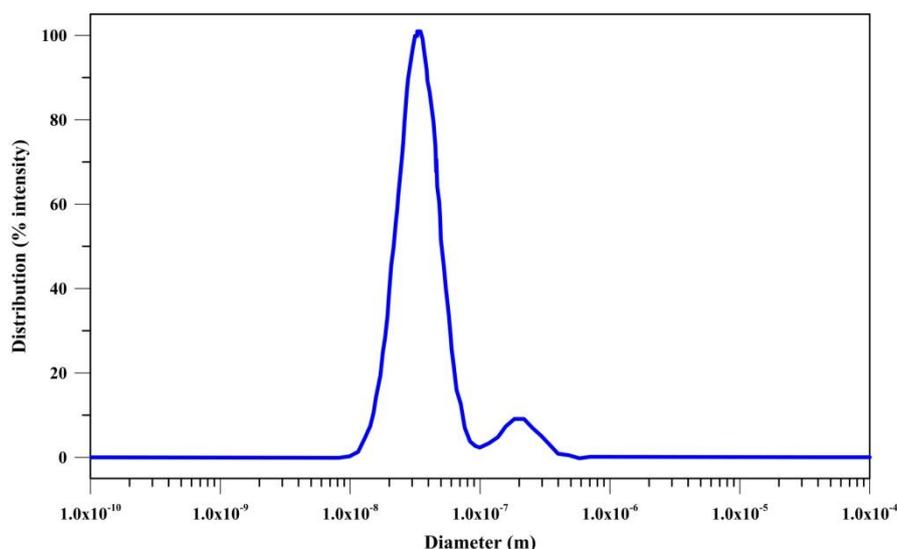


**Figure 3**

FESEM images of the prepared nZVI at different magnifications

#### 3.3. nZVI particle size distribution

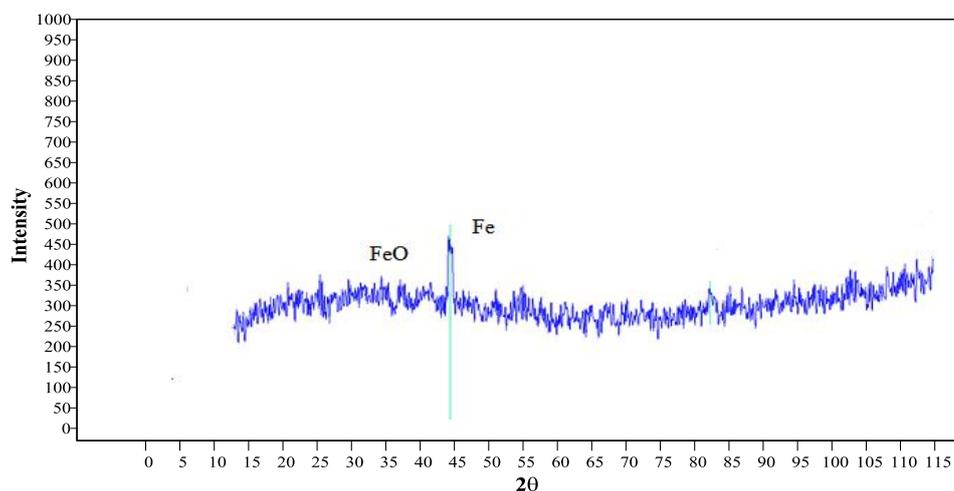
Figure 4 shows the particle size distribution of the nZVI particles. It can be seen that the size of 50% of the particles is 43.3 nm and 90% of the particles are smaller than 80.9 nm. In other words, this work suggests that the nZVI particles are nearly polydisperse with a majority of the particles located within a range of smaller than 80 nm.



**Figure 4**  
Particle size distribution (PSD) of nanoparticles

### 3.4. XRD analysis

The XRD spectrum of the nZVI particles is shown in Figure 5. The broad peak reveals the existence of an amorphous iron phase. The peaks at  $2\theta$  values of  $35.8^\circ$  and  $44.7^\circ$  show the existence of both iron oxide (FeO) and zero-valent iron ( $\alpha$ -Fe) crystalline phase. Investigations showed that the nZVI particles produced by the borohydride reducing agent had iron crystalline phases for  $\text{Fe}^0$ , but the nZVI particles produced by green tea and sorghum bran extracts was amorphous in nature, possibly explaining the low intensity of the diffraction lines of  $\text{Fe}^0$  in this work (Rasheed, et al. 2011; Schrick, et al. 2002).



**Figure 5**  
XRD patterns of the prepared nZVI particles

### 3.5. Hydrocarbon degradation experiments

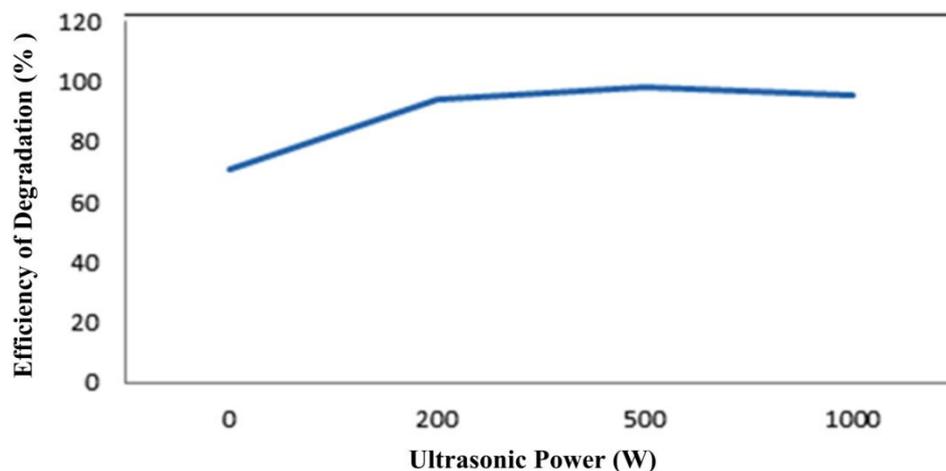
#### 3.6. Effect of ultrasonic power

The application of ultrasonic waves in analytical chemistry is gotten chiefly from acoustic cavitation. Cavitation from ultrasonic waves is the mechanism of transferring the acoustic energy into intense

physical forces within the medium at a localized scale. Cavitation starts with the production of micro-bubbles within the liquid from the rarefaction of the acoustic wave (Sun, et al. 2006; Shih, et al. 2011). Cavitation phenomenon occurs in the presence of solid particles as observed by a change in the symmetry of bubble collapse. When this phenomenon happens beside a particle, the collapse of bubbles is asymmetrically distributed on the particle surface. When cavitation occurs away from the particles, the cavitation collapse is symmetrical and the shock wave can generate turbulence in the solution (Shih, et al. 2011; Wang, et al. 1997). Ultrasound waves have the potential for the remediation of soil and water contaminated with organic and inorganic contaminants (Masciangioli, et al. 2003; Reddy, et al. 2008).

Hoffman et al. reported that ultrasonic treatment, due to oxidation and generating a highly localized temperature and pressure, was effective in the destruction of organic contaminants in water. The breakdown of hazardous compounds into intermediate products could be relatively easy to monitor (Hoffman et al. 2007; Wang, et al. 1997).

Ultrasonic waves not only can degrade pollutants through oxidation and pyrolysis processes in soil, but also move them, through soil washing, to other sources such as water, which is much easier to decontaminate (Hoffman et al. 2007; Zhang, et al. 2011). The effect of ultrasonic power (0, 200 W, 500 W, and 1000 W) on the degradation of hydrocarbons in contaminated soil in the presence of a 0.4 gr.l<sup>-1</sup> solution of nZVI particles and 30 mM H<sub>2</sub>O<sub>2</sub> at 40 °C is shown in Figure 6. It was seen that the efficiency of the degradation rose by increasing sonication power. Around ultrasonic power of 500 W, the degradation reached a maximum of 98.57%. When cavitation occurred, the level of sound pressure at a distance dropped because cavitation took power away from the field. Cavitation could reduce the effective ultrasonic power in soil (Zhang, et al. 2011).



**Figure 6**

Effect of ultrasonic power on the degradation of hydrocarbons in soil in the presence of a 0.4 gr.l<sup>-1</sup> solution of nZVI particles and 30 mM H<sub>2</sub>O<sub>2</sub> at 40 °C

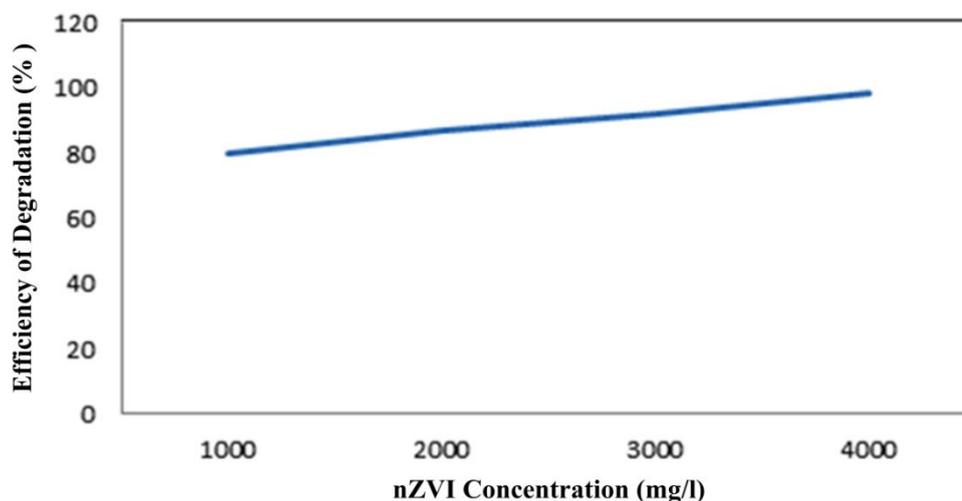
The percentage of the degradation (% degradation) of petroleum hydrocarbons is calculated by Equation 2:

$$\% \text{ degradation} = \frac{(C^0 - C)}{C^0} \times 100 \quad (2)$$

$C^0$  is the initial concentration of petroleum hydrocarbons in soil and  $C$  stands for the final concentration of petroleum hydrocarbons in the treated soil.

### 3.7. Effect of nZVI concentration

The effect of nZVI concentration on the degradation of hydrocarbon contaminants in soil in the presence of 30 mM  $H_2O_2$  at ultrasonic power of 500 W and a temperature of 40 °C is shown in Figure 7. All the experiments were performed with a reaction time of 1 hr.



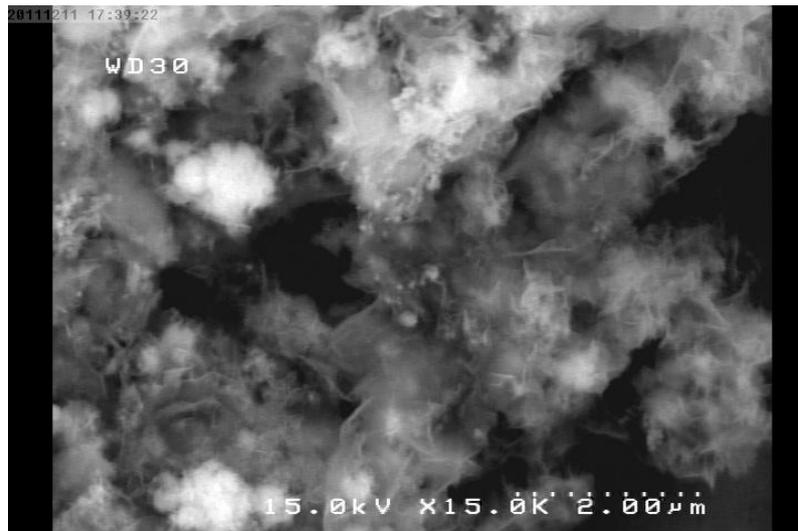
**Figure 7**

Effect of the concentration of nZVI particles on the degradation of hydrocarbon contaminants in soil

The results showed that most of the hydrocarbons contaminated soil was degraded. The degradation of hydrocarbons occurred at the surface of the nanoparticles (Reddy, et al. 2008; Sun, et al. 2006). The transfer of oil into the nZVI solution resulted in extracting oil from the pores of soil and releasing it into the aqueous phase where the oil would be exposed to the reactive surface of the nZVI particles; the FESEM images of the used nZVI particles are shown in Figure 8 (Wang, et al. 1997; Zhang, et al. 2011). The degradation of hydrocarbon chains in soil rose by increasing the nZVI concentration. When the nZVI concentration increased, the concentration of hydrocarbons plummeted and the efficiency of degradation increased from about 80.12% to 98.57%. In fact, the presence of nZVI nanoparticles in the degradation reactions enhanced the efficiency of the degradation.

### 3.8. Effect of $H_2O_2$ concentration

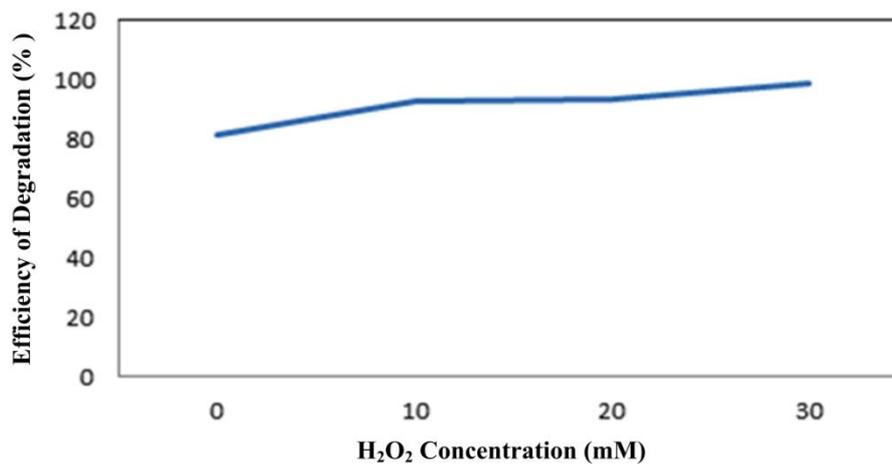
After 30 min of the pretreatment for the reduction of hydrocarbon contaminants with the nZVI particles and ultrasonic wave,  $H_2O_2$  was added at concentrations of 0 mM, 10 mM, 20 mM, and 30 mM to the solution. Hyun moon et al. studied the total organic carbon (TOC) removal efficiency of the simultaneous and sequential nZVI/ $H_2O_2$  processes and they found that the sequential process had higher removal efficiency (Zhang, et al. 2011). This could be attributed to the fact that the nano iron particles were added to the solution in which, because of pre-treatment with reduction reactions and ultrasonic wave, the hydrocarbon chains had already been broken and converted into smaller molecules; these molecules were easier to degrade as the nZVI particles were oxidized into  $Fe^{2+}$  and then OH radicals were generated by  $H_2O_2$ . Hydrogen peroxide played the role of an oxidizing agent in the degradation process (Reddy, et al. 2008; Sun, et al. 2006).



**Figure 8**

The FESEM images of the used nZVI particles

The effect of  $H_2O_2$  concentration was also investigated in the presence of a  $0.4 \text{ gr.l}^{-1}$  solution of the nZVI particles at a temperature of  $40 \text{ }^\circ\text{C}$  and ultrasonic power of  $500 \text{ W}$ . The results are shown in Figure 9. It is clear that by increasing  $H_2O_2$  concentration, the efficiency of degradation increased. This could be ascribed to the fact that when  $H_2O_2$  concentration increased, the concentration of free radicals ( $OH^\bullet$ ) rose in the solution (Sun, et al. 2006). Equations 3, 4, and 5 show the formation of free radicals and the propagation of the degradation reaction.



**Figure 9**

Effect of  $H_2O_2$  concentration on the degradation of hydrocarbon



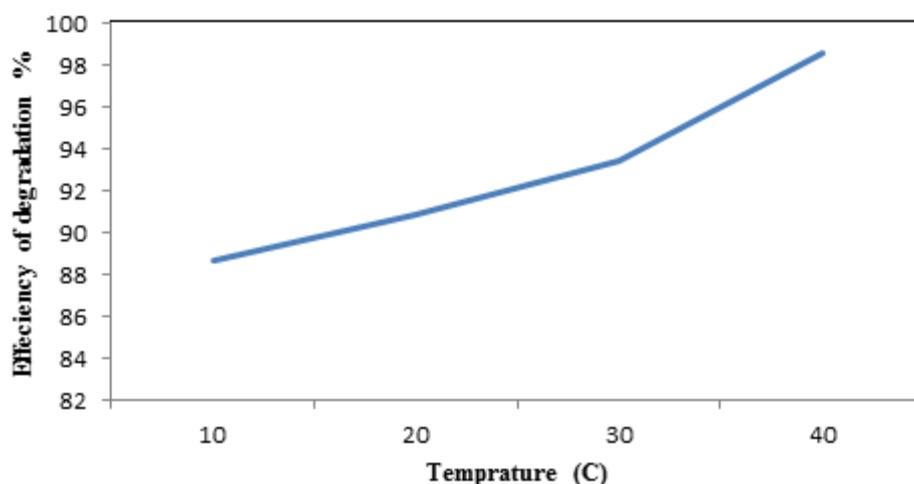
Radical propagation:





### 3.9. Effect of temperature

Temperature effect was studied in the temperature range of 10 °C to 40 °C at ultrasonic power of 500 W in the presence of 30 mM H<sub>2</sub>O<sub>2</sub> and a 0.4 gr.l<sup>-1</sup> solution of the nZVI particles. A gradual enhancement in the removal of oil from soil by the nZVI particles was observed as the temperature was increased from 10 °C to 40 °C. As shown in Figure 10, the approximately similar removal efficiency at 10 °C and 30 °C indicated that the effect of temperature on the degradation of hydrocarbon chains in soil was quiet small. However, rising temperature increased the efficiency of the degradation of hydrocarbon chains in soil.



**Figure 10**

Effect of temperature on the degradation of hydrocarbon contamination in soil

## 4. Conclusions

The aim of this study was to investigate the reactivity of the nano zero valent iron particles synthesized by the assistance of ultrasonication for the degradation of hydrocarbon contamination in soil. Particle size distribution, morphology, and surface composition were characterized using FESEM, XRD, BET, and PSD analysis techniques. The nZVI particles demonstrated some dispersion with a particle size ranging from 40 nm to 80 nm; BET surface area was 35.8 m<sup>2</sup>.gr.<sup>-1</sup>. This work suggests that the nZVI particles were nearly polydisperse with a majority of the particles located within a range of smaller than 80 nm. In the degradation process at increased ultrasonic power, the degradation rate increased to a maximum of around 500 W and then decreased. The degradation of contaminants is increased by increasing the nZVI concentration from about 80.12% to 98.57%. The reactivity of the nZVI particles improved by increasing temperature and H<sub>2</sub>O<sub>2</sub> concentration; nevertheless, the rate of the degradation of contaminants did not change very much at low temperatures. The results indicated that the maximum efficiency of hydrocarbon removal by the nZVI particles was 98.57% and the optimum conditions of degradation were obtained when pH, ultrasonic power, nZVI concentration temperature, and H<sub>2</sub>O<sub>2</sub> concentration were respectively equal to 3.5, 500 W, 0.4 gr.l<sup>-1</sup>, 40 °C, and 30 mM. Some practical applications and suggestions for further work such as finding the optimum conditions of the nano zero-valent iron particles synthesized, studying the effects

of other parameters on the degradation of hydrocarbons in soil, and the environmental benefits and risks of using nano zero-valent iron particles for soil remediation were finally mentioned.

### Acknowledgment

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### Nomenclature

MRI	: Magnetic resonance imaging
PHCs	: Petroleum hydrocarbons
nZVI	: Nano Zero valent iron
PRB	: Permeable reactive barrier
PCBs	: Polychlorinated biphenyls
FESEM	: Field Emission Scanning Electron Microscopy
XRD	: X-ray diffraction

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