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Research Paper

# Applied Analysis of Graphene Nanoparticle in Rotating Disk Reactor to Investigate the Reduction of Pollutants in Industrial Wastewater

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## 1. ABSTRACT

In this research, by using a reactor containing plates covered with graphene nanoparticles and placing it after the anaerobic biological purification unit of the Sari oil refinery facility, it has been tried to reduce the pollution for the next stages of purification by absorbing aromatic substances resulting from the anaerobic biological degradation stage. The results of field emission electron microscope (FE-SEM) analysis showed that the graphene layer on the reactor plates was multilayered. Then, the effect of three parameters of effluent concentration (COD =10, 70, 10, 150 mg/L), pH (5, 7, 9) and temperature (20, 30, and 40°C) on the removal of aromatic compounds, COD, and effluent turbidity were investigated. The results showed that high concentration of COD had better rapid absorption by graphene than its low concentration. Among the three tested temperatures, more absorption was observed at the high temperature. Also, with increasing pH from 5 to 9, the amount of absorption increased. This system showed relatively good performance in absorbing aromatic substances and reducing COD. Optimum conditions for reactor operation were determined as 150 mg/L COD concentration, pH equal to 9, and temperature as 40˚C.

**Keywords:** Sari oil facility wastewater treatment plant, graphene, rotating disk reactor, aromatic compounds, surface absorption.

## 2. INTRODUCTION

The aim of this research is to investigate the performance of graphene as an adsorbent in a rotating disk reactor to remove COD and aromatic substances from the anaerobic treatment of Sari oil facilities. Hence, the effect of three parameters of effluent concentration (COD: 70, 100, 150 mg/L), pH (5, 7 and 9) and temperature (20, 30 and 40 ˚C) on the removal of aromatic compounds, COD and turbidity Effluent will be checked. The use of a rotating disk reactor has the advantage that the contact surface of graphene with wastewater has increased, the need to collect graphene from the effluent is eliminated, and the absorption process will be carried out in a completely controlled and reliable environment in terms of operational conditions. Also, the small space occupation and relatively small initial and operating costs of using the reactor can make it a suitable choice for small industries

## 3. MATERIALS AND METHODS

Graphene used in this research was purchased from ACS Materials, USA with BET surface area of 650 $m^2/g$. and DMF solvent are from MERCK, Germany. The wastewater used in the research was prepared and used from the Sari oil facility and from the second anaerobic treatment outlet of the treatment plant. The reactor designed in this research has a plexiglass body with a height of 50 cm and an internal diameter of 9 cm, a useful volume of 3.9 L and an operational volume of 2 L. A stainless steel rod was considered as its axis, on which three metal discs with a diameter of 6 cm, which can be separated from the axis, were placed.

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### 3-1. Adsorption tests

0.01 g of graphene was dissolved in 10 mL of solvent and then mixed well for 30 minutes and then subjected to ultrasound for 15 minutes. All the obtained solutions were used for coating 4 mica sheets. Due to the brittleness of the mica plates, stainless steel discs were used as holders for these plates inside the reactor, and the mica plates were connected to the metal discs using aquarium glue. In order to investigate the effect of different concentrations on the percentage of aromatic substances removal and COD reduction, the amount of chemical oxygen-demand (mg/L) was used as different concentrations. And for three concentrations of COD: 70, 100, 150 mg/L: experiments were carried out. The parameters pH=7 and T= 40˚C were considered constant. Temperatures of 20, 30 and 40˚C were chosen to test the effect of paratherm on aromatics absorption and COD reduction. The pH=7 and the concentration of COD: 150 mg/L was considered constant. In order to investigate the effect of pH on the percentage of aromatics removal and COD reduction, experiments were conducted at different pH levels of 5, 7, and 9 at a constant temperature of 40°C, and at a constant concentration of 150 mg/L COD. According to the effluent analysis, the pH of the second anaerobic output was equal to 7.1, and $H_2SO_4$ and NaOH were used to make it acidic and alkaline, respectively. An automatic pH meter (pH Meter BASIC 20+ Crison, EU) was used to check pH.

### 3-2. Analysis

APHA 5220 D standard was used to measure COD, which is known as closed reflux. In order to check the degree of removal of aromatic amines from the effluent, ultraviolet-visible spectroscopy was carried out using a spectrophotometer (T80+ UV/VIS Spectrometer PG Instruments Ltd) and it was carried out in the spectrum range of 199 to 900 nanometers by the device. For Fourier Transform Infrared (FT-IR) analysis, using a vector33 device, Bruker Germany, the effluent was placed in an oven for 24 hours at a temperature of 50°C, and after drying, it was analyzed by the KBr tablet method. An automatic AL450T-IR turbidity meter made in England was used at ambient temperature to perform this test. The samples were placed in special glasses for the turbidity meter and the turbidity number was read in NTU units. Field Emission Electron Microscope (W440) (FE-SEM (HITACHI S4160, Japan) was used to perform this analysis at ambient temperature and humidity. The magnification range of the device was from 500 nm to 30 µm and under a voltage of 20 kV.

## 4. RESULTS AND DISCUSSION

### 4-1. Investigating the graphene layer

The results of FE-SEM analysis (Figure 1) showed that the layering was done well. The arrows in this figure show the creases on the graphene surface, which are one of its characteristics. The creases on the surface of the graphene sheet indicate the bonding of the single-layer graphene sheets.

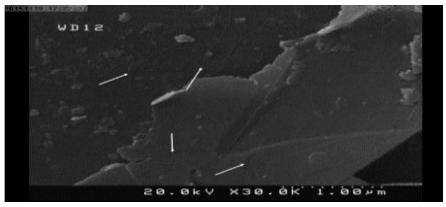


**Figure 1-** FE-SEM image of the surface of mica sheet covered by graphene.

The arrows show the creases on the graphene surface. Ultraviolet-visible spectroscopic analysis was carried out in the wavelength range of 190-900 nm. Since the absorption of aromatic substances occurs in the ultraviolet region and from 200 to 350 spectrum, this area was investigated to investigate the possibility of the presence of aromatic substances in the wastewater [1]. According to this analysis, it was found that there were aromatic substances in the effluent, which were not removed in the previous stages of purification. The highest absorption was observed at the wavelength of 326 nm and this wavelength was used to qualitatively check the removal percentage of aromatic substances.

### 4-2. Investigating the effect of operating parameters on absorption efficiency

The decreasing trend for all concentrations was strong in the first few minutes, but gradually and with the passage of time, this trend takes a steady state. At high concentration, this concentration gradient is very high, and for this reason, the speed of absorption and the rate of absorption are high, but with the decrease in concentration, the strength of the concentration-gradient is reduced and the speed of absorption decreases. The COD reduction percentages for concentrations (70, 100, 150 mg/L) were equal to 57%, 54% and 62%, respectively. The percentage of turbidity reduction for different concentrations were obtained as 2%, 11.5% and 13.9% for concentrations (70, 100, 150 mg/L), respectively.

### 4-3. Temperature

The percentage removal of aromatic substances from the effluent for a temperature of 40 degrees Celsius is equal to 24%, which is much more than the other two temperatures of the test, 20 and 30 ˚C, each with a removal percentage of 13.2% and 13.6%, respectively. It is possible that due to the increase in temperature, the graphene will be swollen and the layers will be separated. This swelling causes more space and as a result more surface area to be created for absorption inside the absorbent internal structure, which causes more large molecules to penetrate inside the graphene and get absorbed in

it. This process is called intermediate growth, which is one of the factors that affects absorption and increases efficiency [2].
The turbidity reduction percentages were 13.9%, 12.2% and 10.8% for temperatures of 20, 30 and 40 ˚C, respectively.

### 4-4. pH

When the of pH increases from 5 to 7, the percentage of removal will increase dramatically. The COD reduction percentage at pH=5 is equal to 46% and at pH=7 this value meets 66%. Due to the presence of $H^+$ ions at low pH and the negativity of the surface charge of graphene, the competition between pollutants and positively charged ions is created and the total absorption is decreased. With the increasing of pH to 7, the amount of positive ions caused by pH is greatly reduced and absorption takes place only for the polluting materials in the wastewater. [3].

## 5. CONCLUSION

The effects of changing each of the concentration, temperature and pH variables on the removal of aromatics in the wastewater were also investigated. At high temperature, high effluent concentration and high pH, the removal rate of aromatics was higher than other tests. The removal percentage obtained for aromatics in optimal conditions was equal to 24.1%, which can be a satisfactory result in relation to the consumed graphene and the load of the wastewater. Examining the ultraviolet-visible absorption spectrum shows that the absorption in the first few minutes happened as surface absorption, which is a fast process, and from 20-30 minutes on, the absorption was carried out by structure and penetration into the graphene layers, which is a slow process. From the analysis of the data, it was found that increasing the concentration and temperature has a positive effect on the adsorbent efficiency and improves COD removal. At high concentrations, the ratio of the effluent to the active surfaces of graphene and the concentration gradient are the promoting factors of absorption, and at high temperatures, the increase in molecular motion and the swelling of graphene layers have this effect. In terms of turbidity changes, the proposed system has reduced turbidity by 38% for the tested concentrations. This reduction in turbidity by absorption on the surface of graphene is partly due to the inability to absorb various chemicals that entered the wastewater during dyeing processes or coagulants such as alum that were used in the previous stages of purification and this The amount of absorption is also related to the aromatics in the wastewater and other possible absorbable substances in it. Optimum conditions for reactor operation were determined as COD: 150 mg/L, pH = 9 and temperature 40˚C.

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