



## Removal of Nickel (II) Ions, Low Level Pollutants, and Total Bacterial Colony Count from Wastewater by Composite Nanofibers Film.

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### Abstract:

This study clarifies the important role of nanofibers produced from polyacrylonitrile after reinforcement with iron nanoparticles ( $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>), to expand the applications of these fibers and to combine their ability to remove Nickel (II) from aqueous solutions with their ability to remove pollutants such as total suspended solids, total nitrogen (TN), total phosphor (TP), chemical oxygen demand, and cyanide as well as killing the bacteria in wastewater. The results of the absorption of Nickel (II) after treatment of the aqueous solution were obtained by an atomic absorption spectrometer type (AAs-7000) and a spectrometer type (Hach DR2800) after treating samples of wastewater obtained pollutant absorption results. The adsorption kinetic parameters results for Nickle (II) proved that the rates of increase in the maximum absorption capacity were 43.27 to 133.5 and from 74.63 to 178.571 mg/g when increasing the initial concentration (10-50 mg/L) for first and second order models. The pH, contact time, electrical conductivity, and initial concentration are good indicators of adsorption efficiency of Nickel (II), the high removal efficiency was 23.96 % at low initial concentration. Increase the reduction rate and TN: TP ratio were significant to increase the reduction rate of the total bacteria up 90 % at 8 hours.

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**Key words:** Aqueous solutions, Total Suspended Solids, Chemical Oxygen Demand, Iron Nanoparticles, Polyacrylonitrile

## **1. Introduction**

Recent scientific research in the field of polymer Nano-composites has turned to electro-spinning technology because of its great importance in the production of Nano-fibers that have unique characteristics such as a surface area to volume ratio in addition to mechanical, crystalline, and magnetic properties [1, 2]. On the other hand, the low particle size plays an important role in obtaining good homogeneity of polymeric solutions and good diffusion of nanoparticles in the polymer matrix with very small quantities, these properties widely studied [3-6]. The low particle size leads to an increase in its applications in medical fields such as drug delivery [7] and engineering applications such as absorption or removal of heavy metal ions from wastewater, especially Nickel (II) ions by control of the particle size and surface modification [8].

Therefore, the use of Nano-fibers is a great alternative to the old separation methods due to their large surface area that increases the level of ion absorption rates compared to other types of materials such as resins, foams, conventional fibers, etc. [9, 10].

In addition, pollution with heavy metal ions has become a major danger due to the rapid increase in global industrial activity [10, 11]. Nickel (II) ~~are~~ is among the pollutants that cause major problems for water due to its toxicity and tendency to bioaccumulation, in addition of to the absorption of Nickel (II) dependent on contact time, solution pH, initial Nickle (II) concentration [12-16]. This pollution has properties that are cumulative in nature and cannot be biodegraded [10]. Water can be purified using the unique, independent magnetic properties process called absorption [17], because electrically spun Nano-fibers have limited functional groups that

remove or absorb certain substances from wastewater solutions [10]. Absorption technology, especially the absorption of heavy metal ions from water, is of high importance, sensitivity, and accuracy, in addition to the low cost [14, 15, 18-20], such as complex operational processes, which needed to less operational costly, and represented by the use of optical sensors to detect Hg (II) ions using chromophores, fluorophores, functional polymers, graphene, proteins and biologically modified nanoparticles [21]. Sun, et al synthesize cyclic amine monomer, 1-acryloyl-2, 2, 5, 5-tetramethyl imidazolidine-4-one (ACTMIO) copolymerized which contain many monomers such as acrylonitrile (AN), methyl methacrylate (MMA), and vinyl acetate (VAC). These materials used an antibacterial such as Escherichia coli [22]. Polyacrylonitrile was used as an antimicrobial activity after blending it with the developed chitosan (N-(2-Hydroxy) propyl-3-trimethylammonium chitosan chloride (HTCC)) and converting it into nanofibers, as the addition of 5% of chitosan works to kill bacteria by 100% [8]. In addition, the polyacrylonitrile Nano-fibers have high surface area and good thermal stability, and were therefore used as a membrane for filtration and bacterial removal [23, 24]. On the other hand, used the polyacrylonitrile nanofibers reinforced by single-walled carbon nanotubes to remove chemical and biological from the graywater pollutants [25]. Anderson and Yu studied the effect of acidic pH and basic pH solutions on bacterial growth. The researchers found that acidic solutions had a slight increase in bacterial growth; while bacteria growth decreased significantly in solutions with pH (5-9) [26], high pH ranges also killed bacteria in wastewater [27]. Ted and co-authors studied the relation between the ratio of total nitrogen (TN) to total phosphor (TP) and growth of bacteria; the authors found when the TN: TP ratio is low, the concentrations of cyanobacteria are high [28]. Increasing of the absorption time lead to decreased the concentrations of polluted

wastewater as chemical oxygen demand (C.O.D), total suspended solid (TSS), and cyanide (CN) [29].

The novelty of this study is that it used the PAN:  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanofibers film as adsorption material with high selectivity and fast detection for nickel (II) from aqueous solution. In addition, it used this composite of nanofibers for the reduction of the low level pollutant concentrations such as chemical oxygen demand (C.O.D), total suspended solid (TSS), and cyanide (CN), and reduction of numbers of bacteria in the real samples of wastewater .

## **2. Experimental Section**

### **2.1 Materials Used.**

Polyacrylonitrile powder (PAN), N, N dimethylformamide (DMF) solvent, and Iron Oxide Nano powder ( $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>) from [30]. GX3007 Nickel (II) nitrate hexahydrate with formula (Ni (NO<sub>3</sub>)<sub>2</sub> · 6H<sub>2</sub>O) supplied from Glentham Life Sciences Limited. Hydroxylamine hydrochloride with formula (NH<sub>2</sub>OH ·HCl) and Sodium carbonate with formula (Na<sub>2</sub> CO<sub>3</sub> ) were supplied from Sigma-Aldrich.

### **2.2 Preparation of solutions**

#### **2.2.1 Preparation of Composites of polymeric solutions**

To prepare the functional groups of the PAN surface for obtaining the best interaction between the polymer chains and  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles, PAN containing the nitrile group was reacted with hydroxylamine hydrochloride and in the presence of sodium carbonate. The reaction shown in Figure 1 was carried out according to the following quantities and conditions:

Add *0.4 g* of PAN powder to *100 ml* of deionized water in a *250 ml* bottle for reacting with *16 g* of hydroxylamine and *12 g* of sodium carbonate at a temperature of

70 ° C for 120 minutes and after the end of the reaction the product is filtered and dried.

A 7 Wt.% of PAN was weighed and dissolved with a solvent DMF and percentage weights of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles ( 1.43, 4.3 , and 7.14 Wt.% ) were added to the composite polymeric solution to use it as a composite of nanofibers by the electrospinning method. The reaction between the PAN and  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles is shown in Figure 1 [31]. The electrospinning condition for production of the composites nanofibers were illustrated [30].

### 2.2.2 Aqueous Solution

The aqueous solution which was prepared in the polymers and petrochemical industries laboratory contained 10, 25, and 50 mg / L of the Nickel (II) nitrate hexahydrate for adsorption of the Nickel (II) by using the composite nanofibers films, according to the following steps:

- Dissolving 4.955 grams of (Ni(NO<sub>3</sub>)<sub>2</sub> · 6H<sub>2</sub>O) in one liter of non-ionic water using a 1000 mL glass volumetric flask to obtain a concentration of 1000 mg / L of the nickel (II) ion.
- Using 100 ml of the above solution and dilute it with one liter of non-ionic water to get a concentration of 100 mg/L.
- Using 10, 25, and 50 ml of a concentration of 100 mg / L and dilute each amount to 1000 ml to obtain concentrations of 10, 25, and 50 mg / L of the nickel (II) ion with a pH range (3.5 -7.5) at room temperature.

### 2.3 Wastewater Source and Sample Characteristics

This study used three samples ~~are~~ taken from the final filtration tanks of the Al-Muaymira site in Al-Hilla City - Iraq before it was discharged into rivers and bodies of water. Table 1 represents the physicochemical analysis of the Al-Muaymira

wastewater - Iraq by using the spectrometer (Hach DR2800) before treatment by composite nanofibers and limits of parameters according International Standards.

## **2.4 Methods**

### **2.4.1 Nano-fibers Fabrication**

Fabrication of the composite nanofibers was achieved by using electro-spun conditions (Nadhim and Habeeb) [20]. In this work, glass slides of (7.5 \*2.5 cm) were used after being weighed and fixed on the surface of the rotary drum collector of the electro-spinning device for use in absorbing nickel (II) Ions from the solutions prepared in (2.2.2) and the wastewater in the (2.3) sections.

### **2.4.2 Adsorption**

The glass slides used in this method after collecting the optimum composites Nano-fibers (as seen in Figure 2 B) was 4.3 Wt. %  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>/ PAN nanoparticles [20] with a weight ( $0.0038 \pm 0.0006$  g), in addition of to the area of collected Nano-fibers was (50\*25 mm). On the other hand, Nickel (II) adsorption process was conducted according to the following steps:

- Immersed the glass slides after depositing the Nano-fibers on them in a solution of 100 mL at concentrations of 10, 25, and 50 mg / L respectively, for period's time as 2,4,6,8 hours.
- After the absorption time is over, the glass slide with Nano-fibers is removed and the aqueous solution is filtered with a filter paper.
- Samples of the aqueous solution are sent to check the pH Meter, the electrical conductivity, and to check the remaining Nickel (II) concentrations after treatment by using atomic adsorption spectroscopy (AAs-7000).

On the other hand, the treatment of the wastewater of the Al-Muaymira site by composite nanofibers takes place according to the following steps:

- Immersed the glass slides after depositing the composite Nano-fibers on them in wastewater samples for *20,40,60,80,100,120 minutes*.
- After the end of the contact time, the glass slides with Nano-fibers are removed from the wastewater and the water is filtered using filter paper.
- Samples are sent to check the pH Meter, electrical conductivity, and measure the physicochemical analysis of the Al-Muaymira wastewater parameters after treatment by composite nanofibers for many periods of time, which represented by the Table 2.

### **2.4.3 Calculating the Total Bacterial Colony Count**

Bacteria and the others pollutants diffused in the real wastewater can be removed by the composite nanofibers according to the following steps:

- Using one millimeter of treated wastewater for each sample according to *2,4,6,8 hour* intervals and placing each sample in the nutrient agar plate.
- The bacteria culture medium (Nutrient agar) is prepared in the biomaterials laboratory at a temperature of *50 °C* so as not to kill bacteria, and then it is placed in a (CRYTE-PURISTER) autoclave.
- The bacteria culture medium is added to the wastewater sample placed in the nutrient agar plate and they are placed in an incubator for 24 hours at a temperature of *37 °C*.
- The total bacteria for each sample is calculated according to the Equation 1:

$$\frac{CFU}{mL} = \frac{no \cdot of \cdot colonies * dilution \cdot factor}{volume \cdot of \cdot culture \cdot plate} \quad (1)$$

Where CFU is a colony-forming unit [32]. Figure 2A represents the nutrient agar plates containing the total bacterial colony count before and after treatment for many times.

## 2.4 Characterization

The removal of nickel (II) ions from aqueous solutions by Atomic Absorption Spectrometer (AAs-7000, Shimadzu- Japan), as well as the determination of low-level pollutants in wastewater such as Chemical oxygen demand (C.O.D), Total suspended solids (TSS), Total Phosphor (TP), Nitrate (NO<sub>3</sub>-2), Nitrite Ion (NO<sub>2</sub>-2), Cyanide (CN), and Total Nitrogen (TN) by the ampoules of (Hach chemical Kit) with a spectrophotometer (Hach DR2800). Additionally, the determination of the PH by (HQ11D Portable pH Meter with Rugged Field Gel pH, Hach) and electrical conductivity of the solutions after treatment is done by HM digital Combo Meter TDS PH EC (COM-300). On the other hand, the preparation (Nutrient agar) to calculate the total bacterial colony count in the treated wastewater for all samples was done by (CRYSTE-PURISTER) Autoclave.

## 3. Results and Discussion

### 3.1 Absorption of Nickel (II) from Aqueous Solutions

#### 3.1.2 Effect of pH Solution and Contact Time

In this study, pH is one of the important factors for the detection of nickel ions in aqueous solution. It was used as an indicator of the absorption efficiency and the amounts of Nickel (II) that were removed from the aqueous solutions for the different initial concentrations. The absorption capacity was calculated at a contact time (t) according to the Equation 2:

$$qt = \frac{Co - Ct}{m} * V \quad (2)$$

Moreover, the absorption capacity at equilibrium state was according to the Equation 3:

$$qe = \frac{Co - Ce}{m} * V \quad (3)$$

Where ( $C_o$ ,  $C_t$ , and  $C_e$ ) represents the initial concentration, concentrations at a time ( $t$ ), and concentration at equilibrium ( $mg/L$ ), while ( $V$ ) represented the volume of the aqueous solution ( $L$ ), and ( $m$ ) the mass of the nanofiber adsorbent ( $g$ ) [11,33]. On the other hand, the removal efficiency (%) can be calculated according to the Equation 4 [14, 34]:

$$RE\% = \frac{C_o - C_t}{C_o} * 100 \quad (4)$$

Where  $RE$  is removal efficiency (%).

Figure 3 shows the Nickel (II) removal efficiency from aqueous solution for many contact times (Figure 3A) and different solution pH (Figure 3B), with the initial concentration range ( $10-50 mg/L$ ). The experimental results proved that the removal efficiency of nickel (II) from aqueous solutions is directly proportional to the increase in the contact time with the nickel nanofibers before reaching the equilibrium state.

The highest percentages of nickel (II) removal are (23.97, 20.708, and 11.714 %) at the periods  $120 min.$  when the initial concentrations of nickel (II) decreased ( $50-10 mg/L$ ). On the other hand, we note that the pH of the aqueous solutions increases with the increase in the contact time, as well as the decrease of the initial concentrations of the aqueous solutions containing nickel ions. The  $pH$  range represents the highest percentage of nickel ions removal at  $pH$  (6.21-7.63). Therefore, aqueous solutions that contain the lowest concentration of nickel ions give the highest removal rate and high selectivity over a wide range of concentrations. These results were agreed with results in previous studies [35,36].

When analyzing the Figure 4, we found that the  $pH$  values of the aqueous solutions after completing the absorption process for all concentrations gradually shift to the basal state with the increase in the contact period accompanied by an increase in the absorption capacity of Nickel (II) on  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>: PAN nanofibers. The large capacity of

Nickle(II) are (63.06, 136.26, and 154.16 mg/g) at the period 120 min ( as seen in Figure 4A) and aqueous solution  $pH$  7.63, 7.2, and 6.21 (as seen in Figure 4B) when increased the initial concentration of Nickle(II) in aqueous solution from 10 mg / L to 50 mg / L ( as seen in Figure 4C) respectively .

The behavior of adsorption capacity as function of initial nickel (II) concentration was agreed with [37]. It is worth noting that the ( $pH \sim 8$ ) has a high absorption capacity [12, 14, 38, 39]. Table 1 summarizes the comparison results of the absorption capacity and the initial concentrations of nickel (II) for this research and the results of the absorption capacity of the composite nanofibers based on the initial concentrations of nickel (II) in aqueous solutions of the previous research, where the increase in the initial concentrations of nickel (II) leads to an increase in the absorption capacity of these ions on the surface of the nanofibers. The absorption capacity also depends on the efficiency of the functional groups of the filler materials, as well as the decrease in the diameter of the nanofibers, which enhances the increase in its surface area. Therefore, the superiority in the performance of composite Nano-fibers, especially at high levels of concentration, the aqueous solutions with high concentrations are closer to the acidic state than the basic state. The competitive adsorption between the available  $H^+$  and the metal ions also hinder the adsorption, but the quantities of free Nickel(II) is more, which increases the possibility of adsorption capacity of Nickel(II) on the surface of nanofibers. This result agreed with the previous studies' results [40, 41].

The electrical conductivity of solutions was defined by the solution's ability to pass electric current, where cations and anions transfer the current. Aqueous solutions vary in the degree of conductivity of electric current and the ionic strength ranges from low conductivity of ultrapure water to high conductivity of concentrated chemical

samples. The behavior of the electrical conductivity of treated aqueous solutions is decreased when increasing the contact time *0-120 min*. (Figure 5A). The minimum electrical conductivity are (4.15, 9.167, 17.73 mS/ cm). In addition, the electrical conductivity decreased with increased solution *pH*, the minimum value of electrical conductivity at ( 7.63,7.2, and 6.21 *pH* ) (as seen in Figure 5 B) found when increasing the initial Nickle (II) concentration in aqueous solution from 10 to 50 mg/L respectively; therefore, conductivity decreases because number of ions per unit volume decreases.

The above results indicated adsorption material with high selectivity and fast detection for nickel (II) from solution [16, 42- 45].

### 3.1.1 Mechanism of Absorption

To verify the absorbance performance of composites of Nano-fibers for removal of nickel (II) from the prepared aqueous solutions (10-50 mg / L) , which have a *pH* (3.5-7.5) and for contact periods with aqueous solutions (0-120 min). Strengthening the polyacrylonitrile matrix with iron ( $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>) nanoparticles increases the surface area of the composite nanofibers and reduces the diameter of the nanofibers, and increased the magnetite; moreover, the mechanism and kinetics of the adsorption process depend on several factors, such as surface morphology and magnetic behavior of adsorption [36,46]: Pseudo-first-order model.

The Lagergren's (pseudo-first-order) rate Equation 5 employed is given by:

$$\frac{dq}{dt} = K_1(q_e - qt) \quad (5)$$

Where  $k_1$  is the rate constant of pseudo-first-order adsorption ( $\text{min}^{-1}$ ),  $q$  is the amount of metal ion adsorbed at various times ( $\text{mg/g}$ ), and  $q_e$  is the amount of metal ion

adsorbed onto adsorbent at equilibrium ( $mg/g$ ). After integrating with the initial conditions, the equation 5 becomes Equation 6 [33,47]:

$$\ln(qe - qt) = \ln qe - K_1 t \quad \text{Pseudo-first-order (Linear form)} \quad (6)$$

All information about the absorption of nickel (II) Ions by Nano-fibers was fitted into the pseudo-second-order adsorption kinetics according to the Equations 7 [11, 48]:

$$\frac{dq}{dt} = K(qe - qt)^2 \quad (7)$$

After integrating the Equation 7 and rearranging, it results in Equation 8 [12, 18, 20]:

$$\frac{t}{qt} = \frac{1}{K_2 qe^2} + \frac{1}{qe} t \quad \text{Pseudo-second-order (Linear form)} \quad (8)$$

Where  $K_2$  represents the second-order absorption rate constant ( $g/mg \cdot min$ ), and  $1/k_2 qe^2$  represents interception of the relationship between ( $t/qt$ ) and contact time ( $t$ ) and the slope ( $1/qt$ ) [48]. The linear fitting plots of adsorption processes according to the pseudo-first-order and pseudo-second order kinetic models are shown in (Figures 6A and Figures 6 B) when using the composites of Nano-fibers, based on the absorption information of Nickel ions referred to in Table 2, which shows the comparison of the adsorption kinetic parameters results for Ni (II) by pseudo-first-order and Pseudo-second-order models between previous studies and this work. The results of previous studies on the mechanism of adsorption of nickel ions on the surface of the PAN:  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanofibers with different initial concentrations and different polymeric nanofibers were used as adsorption materials. Increasing of the correction coefficient with an increase in the initial concentration, the results of this research agree with the results of all previous research, especially when using polyacrylonitrile.

To investigate the mechanism sorption of the Nicle (II) on the surface of PAN:  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanofibers, we estimated the maximum sorption capacity ( $q_{max}$ ) for the pseudo-first-order (Figure 6 A). The  $q_{max}$  represented the interception of the relation between the  $\ln(qe-qt)$  against  $t$  (eq. 6), while the  $q_{max}$  represented the ( $1/slope$ ) of relation between the  $t/qt$  against  $t$  (eq. 8) for Pseudo-second-order model (Figure 6 B).

The increment in the maximum sorption capacity ( $q_{max}$ ) of Nickle (II) on the surface of PAN:  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanofibers was 137.4 % and 139.3% for the pseudo-first-order and Pseudo-second-order models. While there was a slight increase in the rate constant of sorption K1 and K2 when increasing the initial concentration of Nickle (II) in aqueous solution (10-50 mg/L), the correlation coefficients were high ranging from 0.916 to 0.952 for the pseudo-second-order model; therefore, the better results were obtained using the pseudo-second order model [49] .

### 3.2 Physicochemical Analysis of the Wastewater

The pollutants of sewage and groundwater are among the most important determinants of the use of these water sources for the purposes of watering plants or benefiting from them in other fields. Among the important pollutants is totally suspended solids (TSS ), which is an important indicator of the presence of total dissolved solids in water (TDS ) and it consists of organic and inorganic materials that come from mining, crushing, washing materials, etc. They can be removed using micro-membranes and better, Nano-membranes [50]. The reduction efficiency (%) of pollutant in wastewater can be determined according to the equation (3). Table 3 shows the results of spectrometer (Hach DR2800) for the limited concentrations of untreated sewage pollutants of water for three models taken from the (Al-Muaymira) site, in addition to the limits of wastewater parameters according international

standards. From the above table, we notice that the limits of pollutants had low levels of concentration. In this work we used the PAN:  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanofibers for reduction of these concentrations. Table 4 and Figure 7A show a significant decrease in the rates of concentrations of these pollutants in wastewater, and the reduction efficiency of pollutants reduction reached 98.5, 80.26, 98.3, and 64 % for TSS, C.O.D, NO<sub>2</sub><sup>-2</sup>, and CN at 8 hours respectively.

Reduction (%) in COD for this work was higher than the reduction (%) of the COD when using the Ag-Clinoptilolite /Polyethersulfone nanofiber as an absorber material [51]. On the other hand, reduction efficiency % of TSS for this work was higher than the reduction efficiency % of TSS when using the (PAN: Ag) nanofibers or (PVDF: Ag) nanofibers [52]. In addition, cyanide is considered a toxic pollutant. Removing these pollutants means at the same time removing bacteria even if these pollutants are within the minimum limits. The increase of the efficiency reduction of cyanide (CN) and chemical oxygen demand agreed with previous studies [53].

Pollutants such as (Nitrate (NO<sub>3</sub><sup>-2</sup>), TP, and TN) are related to each other in the case of the presence of organic pollutants in wastewater. Because the samples studied have low concentrations of pollutants (TSS and COD), the removal efficiency % is approximately stable with the increase of the Nano-fiber treatment period for some pollutants such as (TP), while the presence of Nano-fibers is negative on the reduction efficiency of the Nitrate (NO<sub>3</sub><sup>-2</sup>).

Because of some physical and chemical techniques such as absorption, ion exchange or reverse osmosis work to transfer pollution instead of removing it, this is what happened in increasing concentrations Nitrate (NO<sub>3</sub><sup>-2</sup>) after the Nano-fiber treatment process [54]. The treated wastewater concentrations by composite nanofibers at 8 hours periods for TSS and COD were  $2 \pm 0.94$  and  $34 \pm 2.52$  (mg/L) while the

permissible limit wastewater stipulated by the World Health Organization (WHO) were 600 and 250 (mg/L) [55] . In addition, the permissible limit wastewater for TSS and COD according to the National Environmental Quality Standard (NEQS) were 150 and 150 mg/L respectively [56]. The electrical conductivity of a solution expresses the ability of a solution to transmit electric current, and this ability increases with the increase in the density or concentration of metal particles or ions that have an electrical conductivity. Figure 7B shows the electrical conductivity of sewage water and its relationship to contact time and solution *pH*. From the Figure, we note that the electrical conductivity of treated water is lower than that of untreated water, and this conductivity decreases with the increase in treatment time. This is evidence of the efficiency of nanofibers used in removing pollutants by reducing the concentrations of these pollutants with an increase in the treatment time accompanied by an increase in the *pH* of wastewater. These results are consistent with the results of another study that used polymers as adsorbents to remove pollutants from wastewater [45].

### **3.3 Reduction of Bacteria**

The researchers used many techniques such as filtration, reverse osmosis, degassing, sedimentation, flocculation, sedimentation and adsorption for the purpose of removing or reducing pollutants from wastewater [57]. The adsorption technology, especially when using highly porous adsorbent materials such as nanofibers, has a significant role in eliminating bacteria because it increases their efficiency by increasing the total surface area of exposure [58]. The bacterial log reduction was calculated by the Equation 9:

$$R\% = (A - B) * 100 \quad (9)$$

Where R is the reduction in CFU, A is the number of bacteria isolated from the inoculated electro spun nanofibers after defined time contact time, and B is the

number of bacteria isolated from the inoculated electrospun nanofibers at zero contact time [24] . The ratio between total nitrogen to total phosphorous (TN: TP) is one of the important indicators of the concentration of total bacteria colony count (CFU/mL). The lower ratio between the two pollutants leads to the higher bacterial concentration in sewage water. This result indicates at the same time an increase in suspended organic matter (TSS) in the water sewage. Table 4 , Figure 7A , and Figure 2A refer to these results, as we notice in the above table that the (TN: TP) ratio increases with the increase in the treatment periods by Nano-fibers, because bacteria cannot resist high alkaline conditions due to the high pH of the treated water with increasing treatment periods [27]. Therefore, we notice a decrease the total bacteria colony count from (3000 to 400 CFU/mL) at 8 hours of treatment (as seen in Figure 2A) accompanied by a significant increase in the reduction efficiency % of suspended organic matter (TSS), these results were in accordance agreed with previous studies [59]. To compare the percentage of bacterial reduction rate of wastewater with that of abatement coliform bacteria to 1340 [CFU/ml], which represents the quality of drinking water by the nanofibers [60], we note that the rate of bacterial reduction by the nanofibers of this research reached 300 CFU/ml at eight hours.

## **Conclusion**

This study explained the efficiency of PAN:  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanofibers for absorbing Nickel (II) from aqueous solutions at low initial concentrations (10-50 mg/L) and reduction the rate of pollutants concentrations such as (TSS, COD, TP, TN, and CN) and bacteria from real wastewater. PAN:  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanofibers have electromagnetic properties and a high surface area that increases the absorption efficiency. The results of the adsorption kinetic parameters for Nickle (II) by pseudo-first-order and Pseudo-

second-order models indicated that the maximum adsorption capacity of Nickel (II) on the surface of PAN:  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanofibers increased with the increasing contact time, solution pH and initial Nickel (II) concentration. In addition, increasing the initial absorption rate in  $K_1$  ( $\text{min}^{-1}$ ) and  $K_2$  ( $\text{g mg}^{-1} \text{min}^{-1}$ ) was dependent on increasing the initial concentrations, and adsorbent material used. For this work and previous studies, the composite nanofibers have low values of adsorption rate compared with others polymeric materials. On the other hand, physicochemical analysis of the Al-Muaymira wastewater - Iraq by using the spectrometer (Hach DR2800) proved that the composite Nano-fibers have a high ability to reduce the pollutants such as (TSS, COD, NO<sub>2</sub>-2 and CN) from wastewater. The increase in the TN: TP ratio with the contact time leads to an enhancement of the nanofiber efficiency to reduce bacteria from the wastewater which is accompanied by a reduction in the electrical conductivity of the wastewater with an increase in the contact time and the *pH*.

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### **Contributions**

Baseem Ali Nadhim: Methodology, investigation, data curation.

Salih Abbas Habeeb: Resources, conceptualization, supervision, writing and editing.

### **Declaration of Interest Statement**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### Conflict of interests

The Author(s) declare(s) that there is no conflict of interest.

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### Figure Captions:

**Fig. 1** represents the hypothesized reaction of hydroxylamine hydrochloride and iron oxide nanoparticles with the PAN nitrile group

**Fig.2** illustrates (A) the nutrient agar plates containing the total bacterial colony count before and after treatment for many times, (B) the FE-SEM image of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>: PAN nanofibers used for treatment the wastewater of the Al-Muaymira site.

**Fig.3** shows the Nickle (II) removal efficiency (%) from aqueous solution (A) for many contact times (min.), and (B) different solution pH, with the initial concentration (10-50 mg/L).

**Fig.4** shows the Nickle (II) adsorption capacity (mg/g) (A) for many contact times (min.) , (B) different solution pH , and (C) the initial concentration (10-50 mg/L ).

**Fig.5** shows the electrical conductivity (mS/cm) before and after adsorption of Nickle (II) from the aqueous solution by composite nanofibers (A) for many contact times (min.) , and (B) different solution pH , with the initial concentration (10-50 mg/L ).

**Fig.6** Adsorption kinetics of Nickle (II) on composite  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>-PAN nanofibers by using (A) the pseudo first- order model which represented by relation between ln (qe-qt) against t, (B) Pseudo-second-order model which is represented by relation t/qt against t, when increasing initial concentrations of aqueous solution (10-50 mg/L).

**Fig.7** (A) Reduction rate percentages of the Al-Muaymira wastewater parameters - Iraq after eight hours' treatment with present composite Nano-fibers film, Parameters such as solution pH and periods contact time as a function of electrical conductivity of wastewater after treatment by  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>-PAN nanofibers.

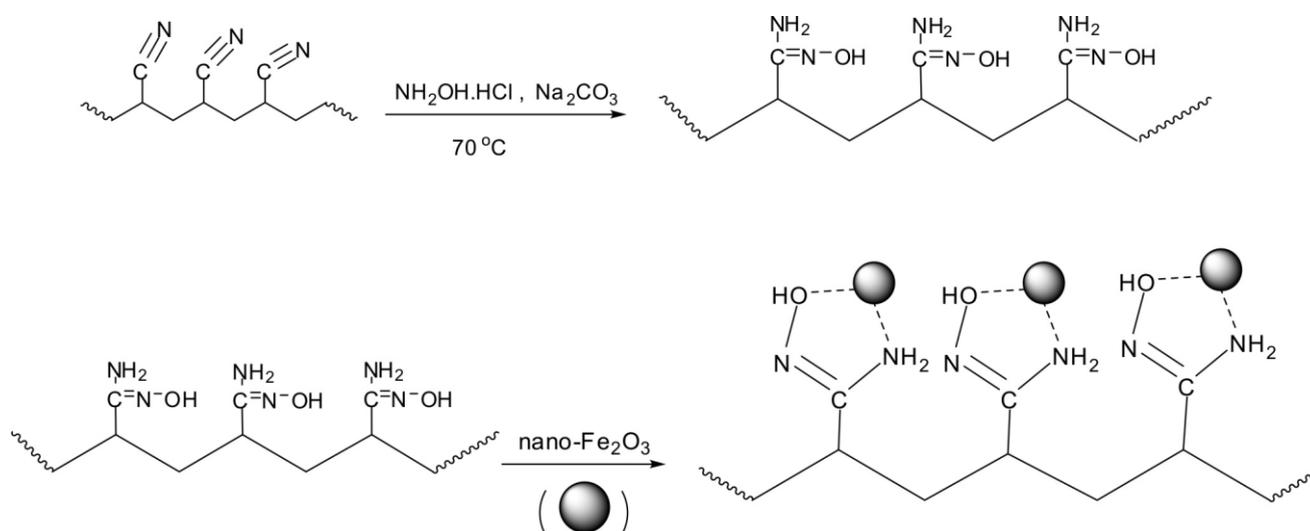
### Table Captions:

**Table1** Comparison of the adsorption capacity, and initial concentrations of Nickle (II) in aqueous solution at 25 °C for previous studies and this work.

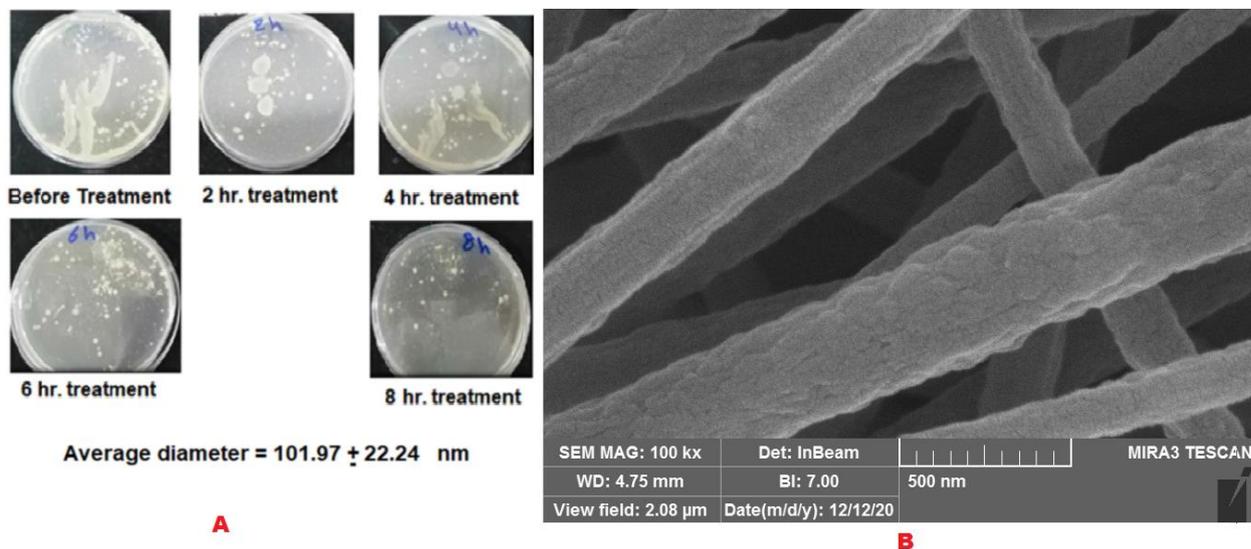
**Table 2** Comparison of the adsorption kinetic parameters results for Nickle (II) by pseudo-first-order and Pseudo-second-order models between previous studies and this work.

**Table 3** shows the physicochemical analysis of the Al-Muaymira wastewater - Iraq by using the spectrometer (Hach DR2800) before treatment and limits of parameters according to International Standards.

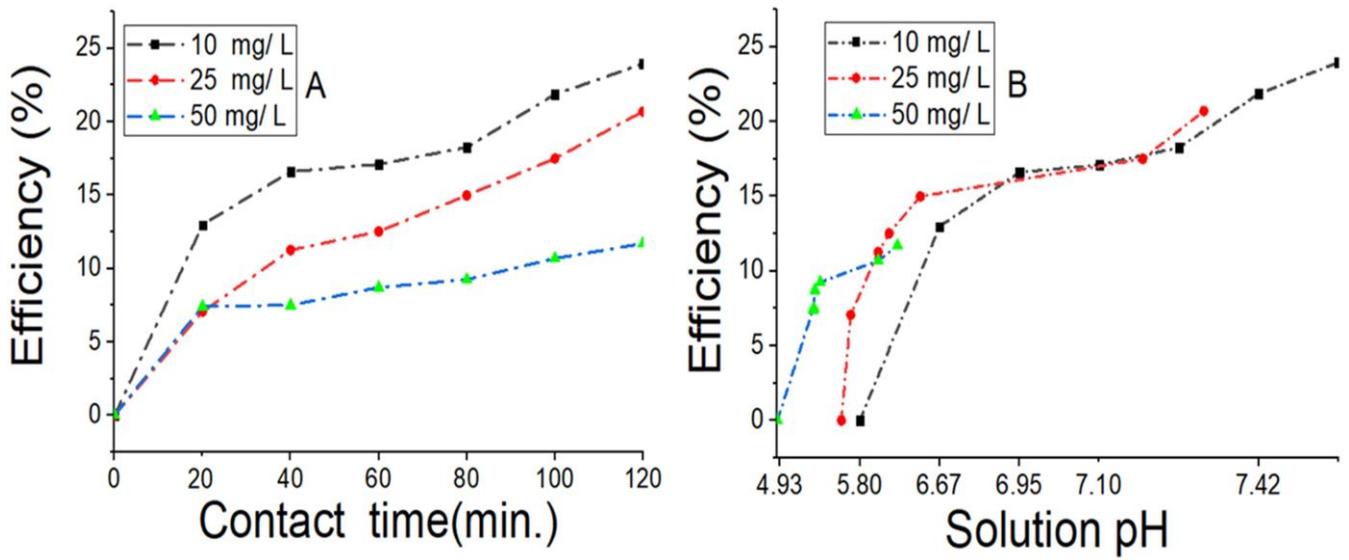
**Table 4** shows the physicochemical analysis of the Al-Muaymira wastewater - Iraq by using the spectrometer (Hach DR2800) after adsorption for many time periods with present composite Nano-fibers film.



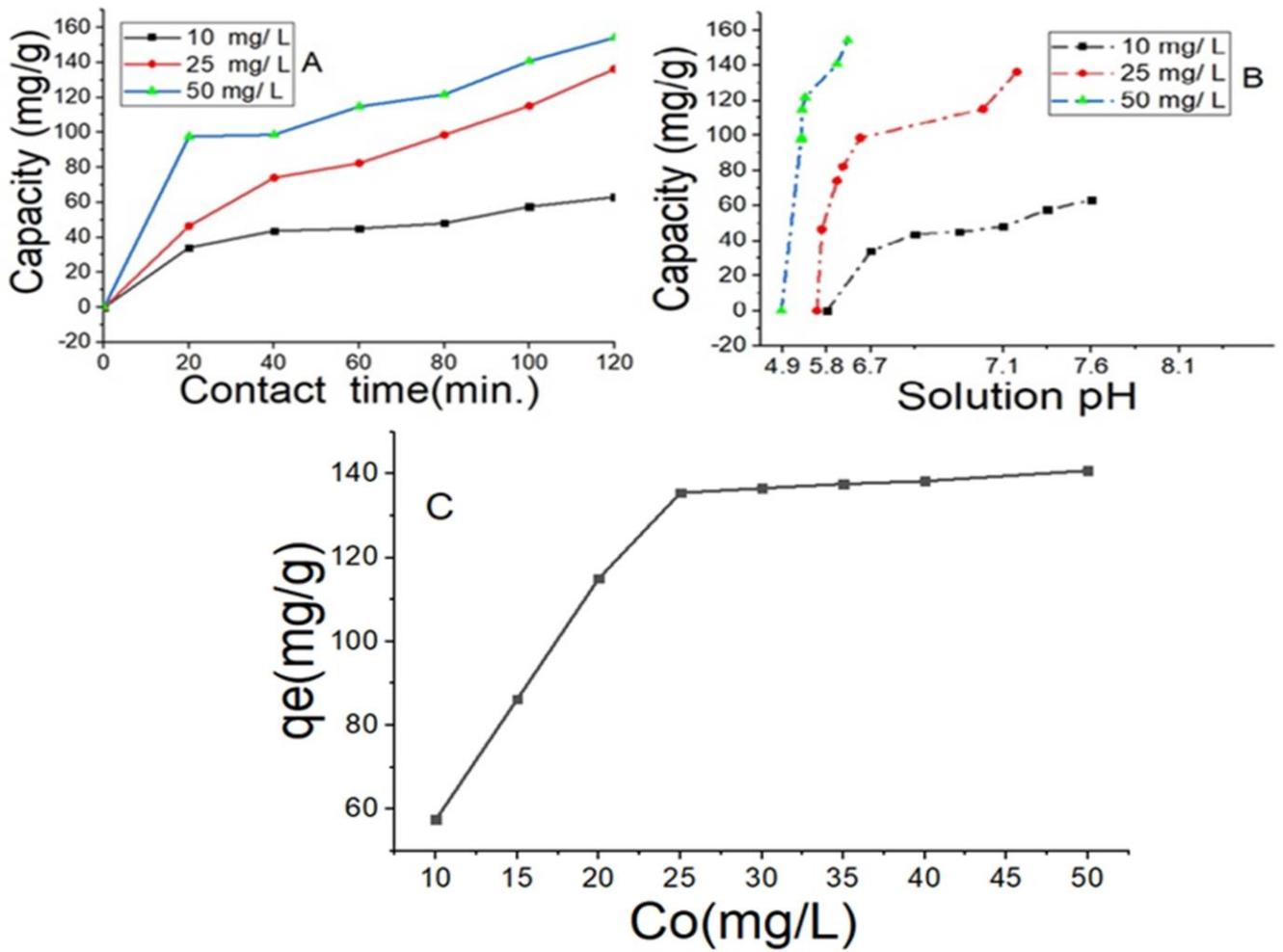
**Fig. 1** represents the hypothesized reaction of hydroxylamine hydrochloride and iron oxide nanoparticles with the PAN nitrile group [31].



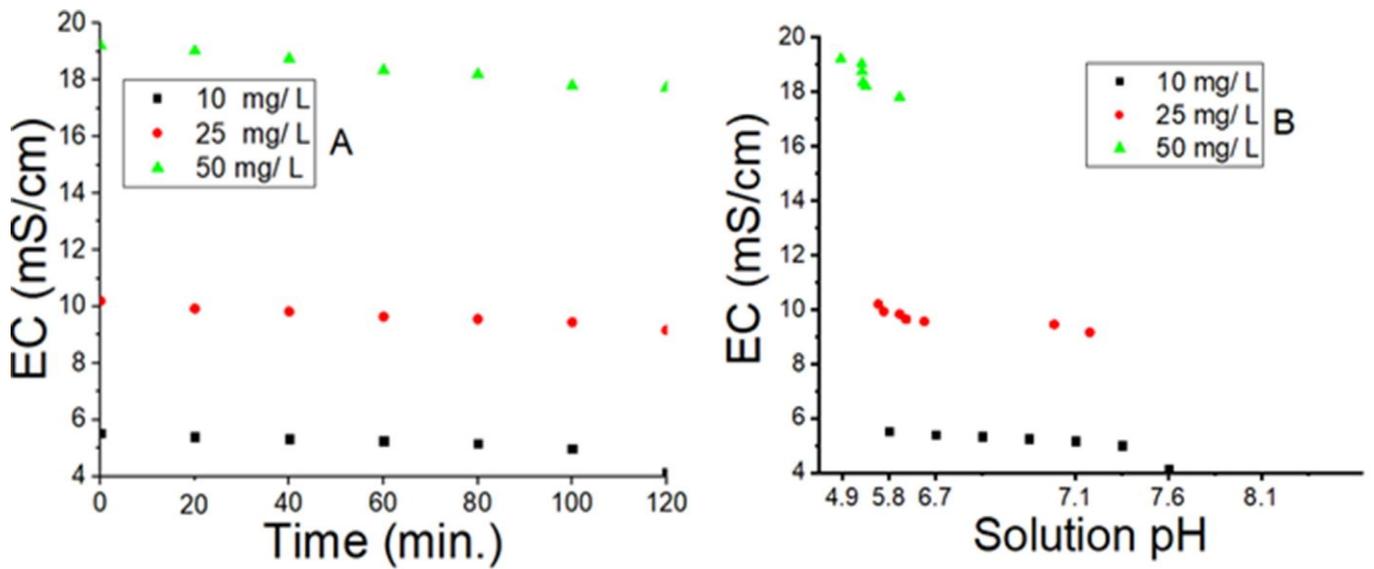
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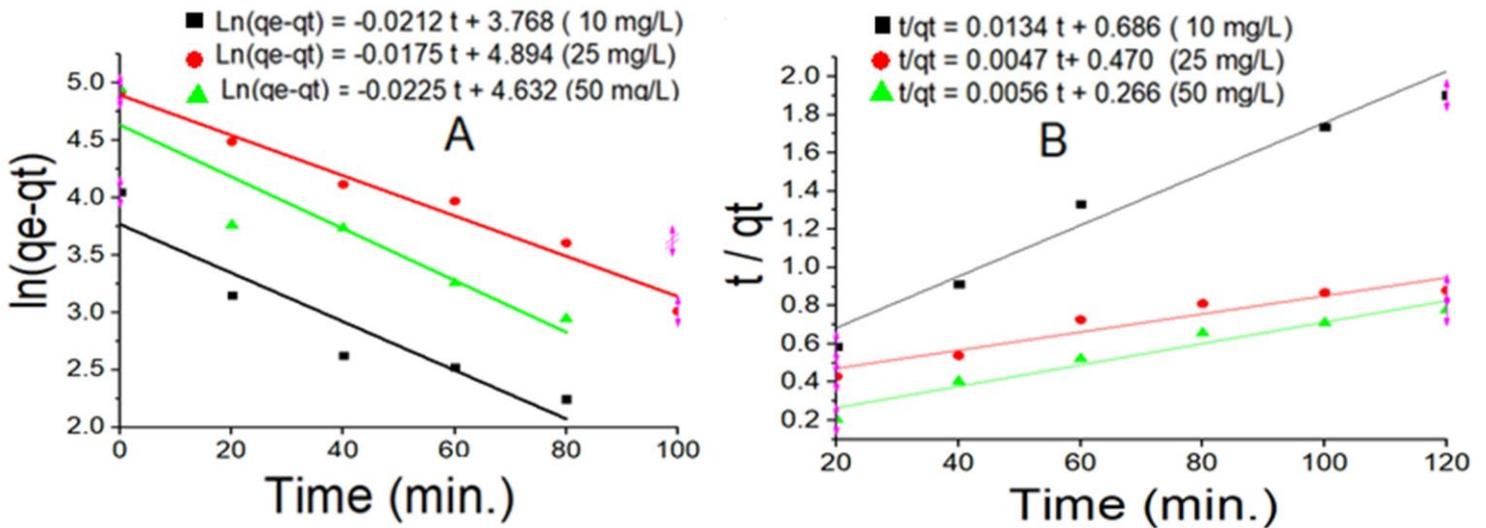
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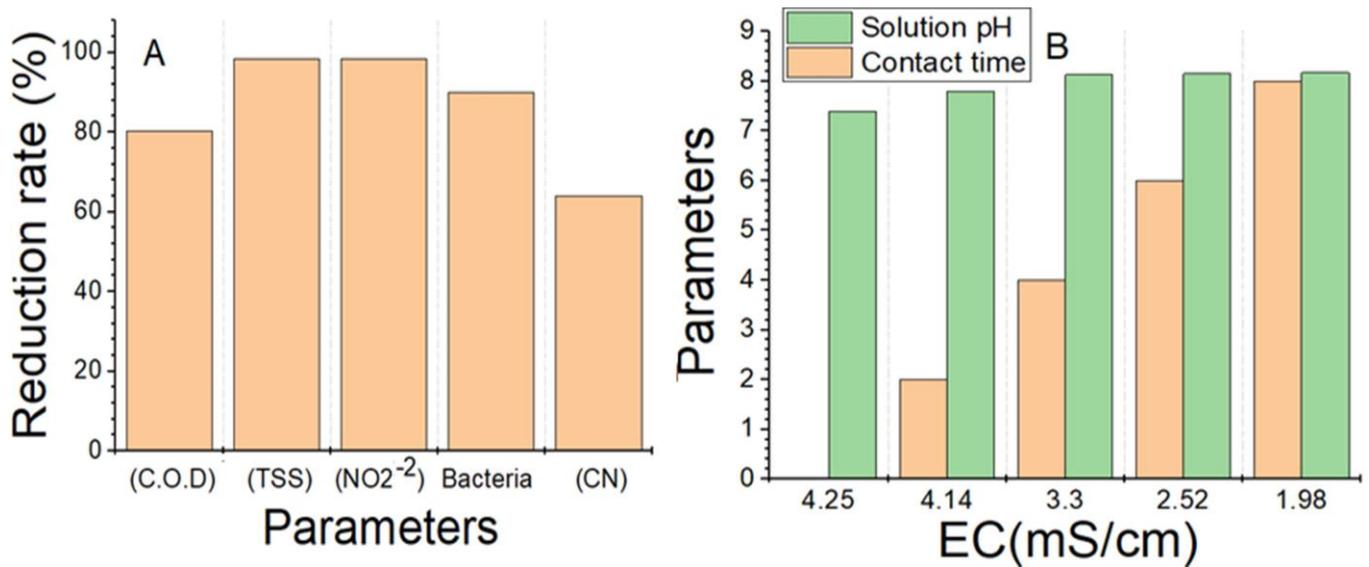
**Fig.4** shows the Nickel (II) adsorption capacity ( $mg/g$ ) (A) for many contact times ( $min.$ ), (B) different solution  $pH$ , and (C) the initial concentration ( $10-50 mg/L$ ).



**Fig.5** shows the electrical conductivity ( $mS/cm$ ) before and after adsorption of Nickle (II) from the aqueous solution by composite nanofibers (A) for many contact times (min.) , and (B) different solution  $pH$  , with the initial concentration ( $10-50\text{ mg/L}$  ).



**Fig.6** Adsorption kinetics of Nickle (II) on composite  $\gamma\text{-Fe}_2\text{O}_3\text{-PAN}$  nanofibers by using (A) the pseudo first- order model which represented by relation between  $\ln (qe-qt)$  against  $t$ , (B) Pseudo-second-order model which is represented by relation  $t/qt$  against  $t$ , when increasing initial concentrations of aqueous solution ( $10-50\text{ mg/L}$ ).



**Fig.7** (A) Reduction rate percentages of the Al-Muaymira wastewater parameters - Iraq after eight hours' treatment with present composite Nano-fibers film, Parameters such as solution pH and periods contact time as a function of electrical conductivity of wastewater after treatment by  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>-PAN nanofibers.

**Table1** Comparison of the adsorption capacity, and initial concentrations of Nickle (II) in aqueous solution at 25 °C for previous studies and this work.

Adsorbent Material	$C_o$ (mg/L)	$q_{max}$ (mg/g)	References
Phosphorylated : polyacrylonitrile nanofibers	150	68.3	[39]
	200	160	
Lewatit Mono Plus SP 112 cation-exchange resin	100	98	[33]
	50	43	
PVA/chitosan nanofibers	13	27	[61]
poly(vinyl alcohol)/silica nanofibers	120	229.9	[62]
Poly ethylene oxide /Chitosan nanofiber	100	175	[63]
	100	84	
cellulose acetate/chitosan nanofibers	200	102	[64]
	300	116	
	400	123	
PAN: $\gamma$ -Fe <sub>2</sub> O <sub>3</sub> nanofibers	10	57.535	This work
	25	135.47	This work
	50	140.733	This work

**Table 2** Comparison of the adsorption kinetic parameters results for Nickle (II) by pseudo-first-order and Pseudo-second-order models between previous studies and this work.

Adsorbent Material	Pseudo-first-order			$R^2$	Ref.
	$C_0$ (mg/L)	$q_m$ (mg g <sup>-1</sup> )	$K_1$ (min <sup>-1</sup> )		
cellulose acetate/chitosan nanofibers	100	75.50	0.54	0.79	[64]
Fe 3 O 4 –coated cellulose acetate/chitosan nanofibers	100	77.47	0.58	0.81	[64]
Phosphorylated : polyacrylonitrile nanofibers	150	51.05	0.0131	0.9497	[39]
PVP/Chitosan/HZSM-5 nanofibers	100	40.34	0.033	0.968	[65]
PAN–TiO2–APTES nanofiber	100	31.5	0.012013	0.9879	[66]
nonwoven composite hydrogel nanofibers	50	47.10	0.0433	0.9988	[37]
PAN: $\gamma$ -Fe2O3 nanofibers	100	94.48	0.0394	0.9998	This work
	10	43.27	0.0212	0.8834	
	25	133.5	0.0175	0.9748	
	50	102.72	0.0226	0.8765	This work
Adsorbent Material	Pseudo-second-order			$R^2$	Ref.
	$C_0$ (mg/L)	$q_m$ (mg g <sup>-1</sup> )	$K_2$ (g mg <sup>-1</sup> min <sup>-1</sup> )		
cellulose acetate/chitosan nanofibers	100	102.20	149×10 <sup>-6</sup>	0.99	[64]
Fe 3 O 4 –coated cellulose acetate/chitosan nanofibers	100	103.40	157×10 <sup>-6</sup>	0.99	[64]
Phosphorylated : polyacrylonitrile nanofibers	150	70.39	0.0028	0.9774	[39]
PVP/Chitosan/HZSM-5 nanofibers	100	46.31	0.0009	0.994	[65]
PAN–TiO2–APTES nanofiber	100	32.0	0.001623	0.9147	[66]
nonwoven composite hydrogel nanofibers	50	46.55	0.2721	0.9670	[37]
PAN: $\gamma$ -Fe2O3 nanofibers	100	93.85	0.3623	0.9276	This work
	10	74.63	0.00026	0.9474	
	25	170.77	0.00010	0.9161	
	50	178.571	0.00032	0.9526	This work

$q_m$ : maximum sorption capacity

**Table 3** shows the physicochemical analysis of the Al-Muaymira wastewater - Iraq by using the spectrometer (Hach DR2800) before treatment and limits of parameters according to International Standards.

Parameters	Limits Before Treatment	Limits according International Standards	References
(C.O.D)	150-154 (mg/L)	1100 120	[67] [68]
(TSS)	129-135 (mg/L)	150 800	[56] [67]
(TP)	2.9-3.4 (mg/L)	25 10 5	[67] [69] [68]
(NO <sub>3</sub> <sup>-2</sup> )	0.7-0.8 (mg/L)	3 50	[70] [68]
(NO <sub>2</sub> <sup>-2</sup> )	0.567-0.587 (mg/L)	3	[70]
(CN)	0.045-0.09 (mg/L)	0.2 2	[67] [69]
(TN)	3.78-4.5 (mg/L)	20 100	[70] [67]
Collection of bacteria	3000-3600 (CFU/ml)	300 CFU/mL 650 CFU/mL	[71] [72]
EC	4.25 (mS/cm)	30 (mS/cm)	[71]
pH	7.38-8.15	6.5-8.5	[67 , 68,73]

International Standards as (Law 93/1962: Law 93/1962 for Discharge of Waste waters provided by food and agriculture organization (FAO), PML: Permissible Maximum Limit in wastewater, world health organization (WHO) ), discharged to national waterbodies, c.o.d= chemical oxygen demand; tp = total phosphor; cn = cyanide; tn= total nitrogen; tss = total suspended solid , CE =electrical conductivity .

**Table 4** shows the physicochemical analysis of the Al-Muaymira wastewater - Iraq by using the spectrometer (Hach DR2800) after adsorption for many time periods with present composite Nano-fibers film.

Parameters	Pollutant Concentrations (mg/L) at many absorption times				
	Initial time	2 hr.	4 hr.	6 hr.	8 hr.
(C.O.D)	152 ±2	39±1.53	70±2.52	32±3	34±2.52
(TSS)	133±3.5	14.53	6±1.54	3±0.36	2±0.94
(TP)	3.1±0.25	3.47±0.49	3.12±0.04	3.0±0.36	3.23±0.5
Nitrate (NO <sub>3</sub> <sup>-2</sup> )	0.7±06	1.4±0.055	1.4±0.06	1.4±0.04	1.4±0.038
Nitrite Ion (NO <sub>2</sub> <sup>-2</sup> )	0.587	0.024±0.003	0.009±0.0037	0.014±0.003	0.01±0.0053
(CN)	0.05	0.009±0.007	0.016±0.0025	0.021±0.002	0.018±0.005
(TN)	2.0±0.37	2.3±0.284	2.1±0.153	2.2±0.277	2.5±0.223
TN:TP Ratio	0.645	0.66	0.67	0.73	0.77
Collection of bacteria (cfu/ml)	3000	2000	1400	800	300
EC (mS/cm)	4.25	4.14	3.3	2.52	1.98

CFU: Colony Forming Unit & cfu/ml calculation, EC: Electrical conductivity

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