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Removal of nickel(II) ions, low-level pollutants, and total bacterial colony count from wastewater by composite nanofiber film

S.A. Habeeb* **and B.A. Nadhim**[1]

Department of Polymer and Petrochemical Industries Engineering College of Materials Engineering, University of Babylon, Al Hilla-51001, Iraq.

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Abstract. This study elaborates on the important role of nanofibers produced from polyacrylonitrile after reinforcement with iron nanoparticles (γ-Fe_2O_3). The objective is to expand the applications of these fibers and combine their ability to remove nickel(II) from aqueous solutions with the ability to not only remove pollutants such as total suspended solids, Total Nitrogen (TN), Total Phosphor (TP), chemical oxygen demand, and cyanide but also kill the bacteria in wastewater. The absorption results for nickel(II) following the aqueous solution treatment were obtained by an atomic absorption spectrometer type (AAs-7000) and then, a spectrometer type (Hach DR2800) managed to obtain pollutant absorption results after treating wastewater samples. The results of adsorption kinetic parameters for nickel(II) proved that the rates of increase in the maximum absorption capacity ranged from 43.27 to 133.5 and from 74.63 to 178.571 mg/g when increasing the initial concentration (10–50 mg/L) for the first and second-order models. pH, contact time, electrical conductivity, and initial concentration represented good indicators of adsorption efficiency for nickel(II), and the high removal efficiency was 23.96% at a low initial concentration. Intensification of the reduction rate and TN:TP ratio appeared to be significant enough to increase the removal rate of the total bacteria by 90% in 8 h.

1. Introduction

Recent scientific research on polymer nano-composites has given rise to electro-spinning technology which is crucial to the production of nanofibers. Nanofibers enjoy unique characteristics such as surface area to volume ratio as well as mechanical, crystalline, and magnetic properties [1,2]. Their low particle size plays an important role in obtaining good homogeneity for polymeric solutions and favorable diffusion of nanoparticles in the polymer matrix with very small quantities; of note, these properties have been widely studied [3–6]. The mentioned low particle size extends the applicability of nanofibers to medical fields, e.g., drug delivery [7], and diversifies engineering applications, e.g., absorption or removal of heavy metal ions, especially nickel(II) ions, from wastewater through particle size controlling and surface modification [8].

1. *Present address: Department of Ceramics and Building Materials, College of Materials Engineering, University of Babylon, Al Hilla-51001, Iraq.*
*. *Corresponding author.*
E-mail address: drsaleh.abbas@uobabylon.edu.iq (S.A. Habeeb)

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Therefore, backed by their large surface area, nanofibers represent a viable alternative to conventional separation methods and they increase 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 turned into a major threat due to the rapid increase in global industrial activity [10,11]. Nickel(II) is among the pollutants that causes major problems for water due to its toxicity and tendency for bioaccumulation as well as nickel(II) absorption, which is dependent on contact time, solution pH, and initial nickle (II) concentration [12–16]. This pollution is cumulative in nature and cannot be biodegraded [10]. Water can be purified through absorption, which is a unique and independent magnetic property [17], because electrically spun nanofibers have limited functional groups that remove or absorb certain substances from wastewater solutions [10]. Absorption technology, especially absorption of heavy metal ions from water, is of high importance, sensitivity, and accuracy and is quite inexpensive [14,15,18–20]. It is obvious that relevant complex operational processes should be less costly in operation and utilize optical sensors to detect Hg(II) ions using chromophores, fluorophores, functional polymers, graphene, proteins, and biologically modified nanoparticles [21]. Sun et al. synthesized cyclic amine monomer and 1-acryloyl-2, 2, 5, 5-tetramethyl imidazolidine-4-one (ACTMIO) copolymerized, containing many monomers such as acrylonitrile (AN), methyl methacrylate (MMA), and vinyl acetate (VAC). These materials use an antibacterial like Escherichia coli [22]. Polyacrylonitrile was employed to serve 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 5% addition of chitosan suffices to kill bacteria by 100% [8]. In addition, polyacrylonitrile nanofibers enjoy a high surface area and good thermal stability, and they have, therefore, been used as a membrane for filtration and bacterial removal [23,24]. On the other hand, the polyacrylonitrile nanofibers reinforced by single-walled carbon nanotubes were used to remove chemical and biological substances from graywater pollutants [25]. Anderson and Yu studied the effect of acidic pH and basic pH solutions on bacterial growth. They found that acidic solutions could accelerate bacterial growth, while bacteria growth decreased significantly in solutions with pHs 5–9 [26]. High pHs killed bacteria in wastewater [27]. Harris et al. studied the relation between the ratio of Total Nitrogen (TN) to Total Phosphor (TP) and growth of bacteria. They found when the TN:TP ratio was low, the cyanobacteria concentrations were high [28]. Increasing the absorption time would lead to the reduced concentration of polluted wastewater as Chemical Oxygen Demand (COD), Total Suspended Solid (TSS), and Cyanide (CN) [29].

This study makes two contributions as part of its novelty. First, the Polyacrylonitrile (PAN): γ-Fe_2O_3 nanofiber film is used as an adsorption material with high selectivity and fast detection for nickel(II) from aqueous solution. In addition, this composite of nanofibers is used to reduce low-concentration pollutants such as COD, TSS, and CN and to decrease the number of bacteria in real wastewater samples.

2. Experimental section

2.1. Materials

PAN powder, N, N-dimethylformamide (DMF) solvent, and iron oxide nano-powder (γ-Fe_2O_3) were borrowed from the referenced study [30]. GX3007 nickel(II) nitrate hexahydrate with formula ($Ni(NO_3)^{-2}.6H_2O$) was supplied from Glentham Life Sciences Limited. Hydroxylamine hydrochloride with formula ($NH_2OH.HCl$) and sodium carbonate with formula (Na_2CO_3) 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 γ-Fe_2O_3 nanoparticles, PAN containing the nitrile group reacted with hydroxylamine hydrochloride in the presence of sodium carbonate. The reaction shown in Figure 1 occurred under the following conditions and quantities.

0.4 g of PAN powder was added 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. At the end of the reaction, the product was filtered and dried.

7 Wt.% PAN was weighed and dissolved with a solvent DMF, and the percentage weights of γ-Fe_2O_3 nanoparticles (1.43, 4.3, and 7.14 Wt.%) were added to the composite polymeric solution for use as a composite of nanofibers by the electrospinning method. The reaction between the PAN and γ-Fe_2O_3 nanoparticles is shown in Figure 1 [31]. The electrospinning condition for the production of composite nanofibers was presented in [30].

2.2.2. Aqueous solution

The aqueous solution prepared in the laboratory associated with polymer and petrochemical industries contained 10, 25, and 50 mg/L of nickel(II) nitrate hexahydrate for adsorption of nickel(II) using composite nanofiber films. The corresponding steps are as follows:

- Dissolving 4.955 g of ($Ni(NO_3)^{-2}.6H_2O$) in one liter of non-ionic water using a 1000 mL glass volumetric

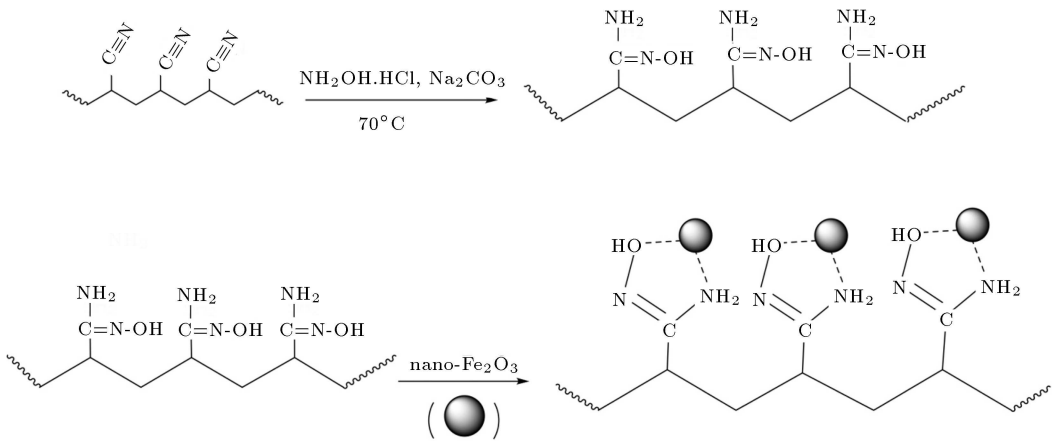

Figure 1. The hypothesized reaction of hydroxylamine hydrochloride and iron oxide nanoparticles with the PAN nitrile group.

Table 1. Comparison of the adsorption capacity, and initial concentrations of nickel(II) in aqueous solution at 25°C in previous and current studies.

Adsorbent material	Co (mg/L)	q_{max} (mg/g)	References
Phosphorylated: Polyacrylonitrile nanofibers	150	68.3	[39]
	200	160	[33]
Lewatit mono plus SP 112 cation-exchange resin	100	98	
	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]
Cellulose acetate/Chitosan nanofibers	100	84	[64]
	200	102	
	300	116	
	400	123	
PAN: γ-Fe_2O_3 nanofibers	10	57.535	This work
	25	135.47	This work
	50	140.733	This work

flask to obtain a concentration of 1000 mg/L of nickel(II) Ion;

- Using 100 ml of the above solution and diluting 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 diluting each amount to 1000 ml to obtain nickel(II) ion concentrations of 10, 25, and 50 mg/L with the pH range of 3.5 – 7.5 at room temperature.

2.3. Wastewater source and sample characteristics

This study used three samples from the final filtration tanks of the Al-Muaymira site in Al-Hilla city, Iraq before being discharged into rivers and water bodies. Table 1 represents the physicochemical analysis of Al-Muaymira wastewater in Iraq using a spectrometer (Hach DR2800) before treatment by composite nanofibers and limits of parameters according to International Standards.

2.4. Methods

2.4.1. Nano-fibers fabrication

The composite nanofibers were fabricated under electro spun conditions [20]. In this work, glass slides of 7.5 × 2.5 cm were used after being weighed and fixed on the surface of a rotary drum collector of the electrospinning device for use in absorbing nickel(II) ions from the solutions prepared in Subsection 2.2.2 and the wastewater in Subsection 2.3.

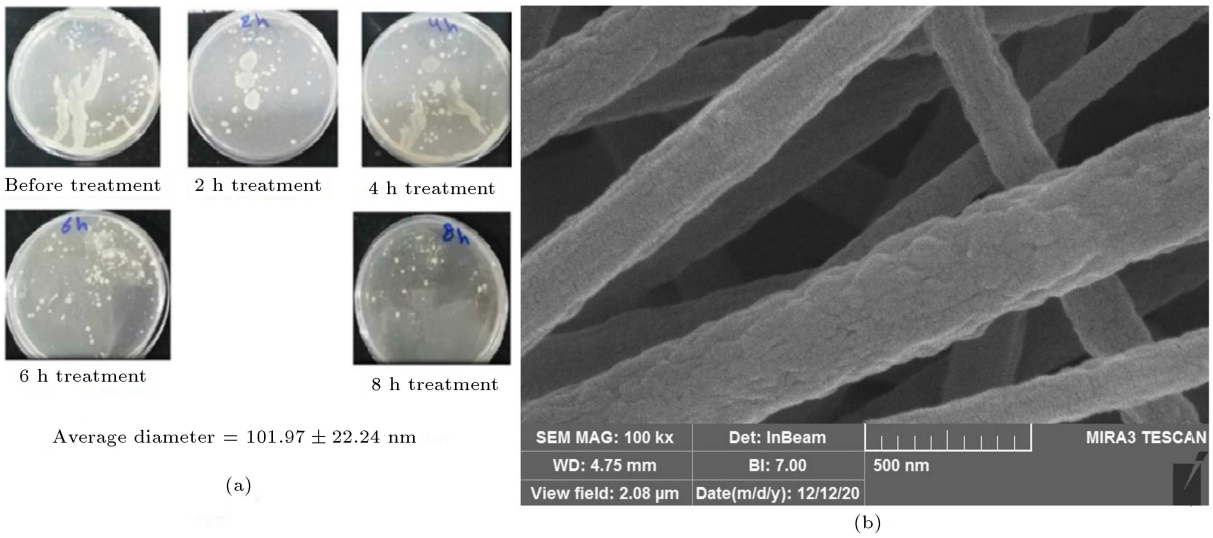

Figure 2. (a) The nutrient agar plates containing the total bacterial colony count before and after applying the treatment in many time periods; (b) The FE-SEM image of γ-Fe_2O_3: PAN nanofibers used for treating the wastewater at Al-Muaymira site.

2.4.2. Adsorption

Following the collection of optimum composite nanofibers (as seen in Figure 2(b)), the glass slides used in this method included 4.3 Wt.% γ-Fe_2O_3/PAN nanoparticles [20] with a weight of 0.0038 ± 0.0006 g, in addition to the 50×25 mm area of collected nanofibers. Furthermore, nickel(II) adsorption process was carried out through the following steps:

- Immersing glass slides followed by nanofiber deposition in a solution of 100 mL at concentrations of 10, 25, and 50 mg/L for 2,4,6,8 h;
- At the end of the absorption, the glass slide with nanofibers 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 the remaining nickel(II) concentrations after treatment by using atomic adsorption spectroscopy (AAS-7000);

Moreover, the treatment of the wastewater at Al-Muaymira site by composite nanofibers takes place according to the following steps:

- Immersing the glass slides after depositing the composite nanofibers on them in wastewater samples for 20,40,60,80,100,120 minutes;
- At the end of the contact time, the glass slides with nanofibers are removed from the wastewater and the water is filtered using a filter paper;
- Samples are sent to check the pH and electrical conductivity and to conduct the physicochemical analysis of the Al-Muaymira wastewater parameters after treatment by composite nanofibers at different times, as represented in Table 2.

2.4.3. Calculating the total bacterial colony count

Bacteria and other 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 h intervals and placing each sample in the nutrient agar plate.
- The bacteria culture medium (Nutrient agar) is prepared in the biomaterial laboratory at a temperature of 50°C so as not to kill bacteria. Then, it is placed in a CRYSTE-PURISTER autoclave.
- The bacteria culture medium is added to the wastewater sample placed in the nutrient agar plate and placed in an incubator for 24 h at a temperature of 37°C.
- The total number of bacteria for each sample is calculated using Eq. (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 2(a) represents nutrient agar plates containing the total bacterial colony count before and after applying the treatment in many time periods.

2.5. Characterization

This section centers on the removal of nickel(II) ions from aqueous solutions by Atomic Absorption Spectrometer (AAS-7000, Shimadzu-Japan) as well as the determination of low-concentration pollutants in wastewater such as COD, TSSs, TP, Nitrate $(NO_3)^{-2}$, nitrite ion $(NO_2)^{-2}$, CN, and TN by the ampules (Hach chemical Kit) with a spectrophotometer (Hach DR2800). In addition, the pH is determined by HQ11D

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

		Pseudo-first-order			
Adsorbent material	**CO (mg/L)**	q_m **(mg g^{-1})**	K_1 **(min^{-1})**	R^2	**Ref.**
Cellulose acetate/Chitosan nanofibers	100	75.50	0.54	0.79	[64]
Fe_3O_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-TiO_2-APTES nanofiber	100	31.5	0.012013	0.9879	[66]
Nonwoven composite hydrogel nanofibers	50	47.10	0.0433	0.9988	[37]
	100	94.48	0.0394	0.9998	
PAN: γ-Fe_2O_3 nanofibers	10	43.27	0.0212	0.8834	This work
	25	133.5	0.0175	0.9748	This work
	50	102.72	0.0226	0.8765	This work
		Pseudo-second-order			
Adsorbent material	**CO (mg/L)**	q_m **(mg g^{-1})**	K_2 **(g mg^{-1}min^{-1})**	R^2	**Ref.**
Cellulose acetate/chitosan nanofibers	100	102.20	149×10^{-6}	0.99	[64]
Fe_3O_4-coated cellulose acetate/Chitosan nanofibers	100	103.40	157×10^{-6}	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-TiO_2-APTES nanofiber	100	32.0	0.001623	0.9147	[66]
Nonwoven composite hydrogel nanofibers	50	46.55	0.2721	0.9670	[37]
	100	93.85	0.3623	0.9276	
PAN: γ-Fe_2O_3 nanofibers	10	74.63	0.00026	0.9474	This work
	25	170.77	0.00010	0.9161	This work
	50	178.571	0.00032	0.9526	This work

Note: q_m: maximum sorption capacity.

Portable pH meter with Rugged Field Gel pH, Hach, and electrical conductivity of the solutions after treatment via HM digital Combo meter TDS pH EC (COM-300). Furthermore, 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

3.1.1. Effect of pH solution and contact time

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

$$qt = \frac{Co - Ct}{m} * V. \tag{2}$$

Moreover, the absorption capacity in an equilibrium state is determined via Eq. (3):

$$qe = \frac{Co - Ce}{m} * V, \tag{3}$$

where (Co, Ct, and Ce) represent the initial concentration, concentrations at a time (t), andconcentration in equilibrium (mg/L), while (V) represents the volume of the aqueous solution (L), and (m) the mass of the nanofiber adsorbent (g) [11,33].

Moreover, the removal efficiency (%) can be calculated using Eq. (4) [14,34]:

$$RE(\%) = \frac{Co - Ct}{Co} * 100, \tag{4}$$

where RE is removal efficiency (%).

Figure 3 shows the removal efficiency for nickle(II) from aqueous solution at many contact times (Figure 3(a)) and different solution pHs (Figure 3(b)), with the initial concentration range of 10–50 mg/L. The experimental results proved that nickel(II) removal efficiency 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% in a period of 120 min when the initial concentrations of nickel(II) are reduced (50–10 mg/L). Moreover, the pH of the aqueous solutions increases with increase in the contact time as well as decrease in the initial concentration of the aqueous solutions containing nickel ions. The pH range represents the highest removal percentage of nickel ions at

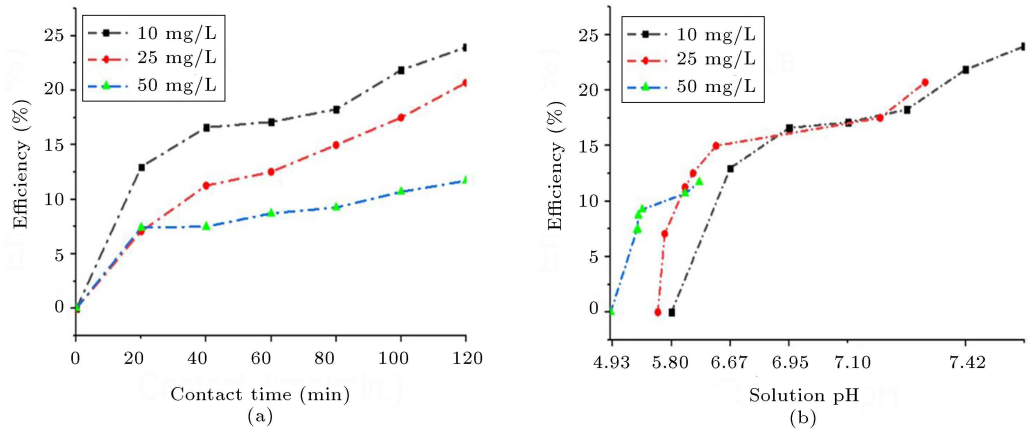

Figure 3. Nickel(II) removal efficiency (%) from aqueous solution (a) for many contact times (min) and (b) different solution pH, with the initial concentration (10–50 mg/L).

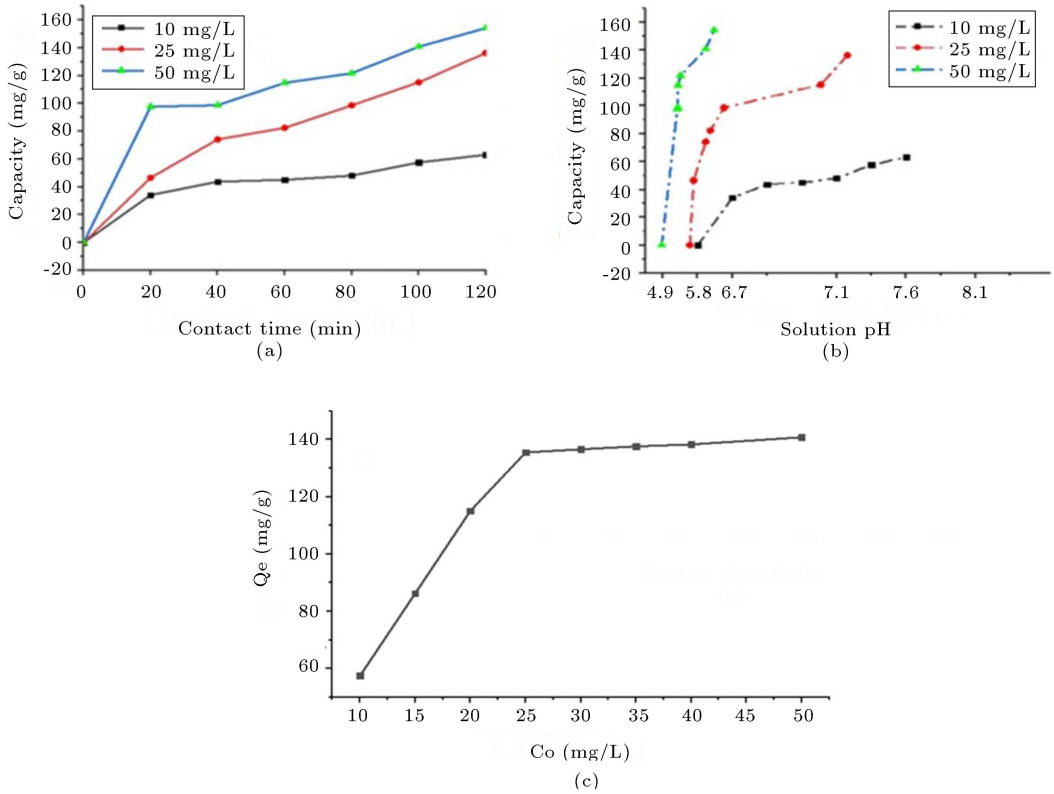

Figure 4. Nickel(II) adsorption capacity (mg/g) (a) for many contact times (min), (b) different solution pHs, and (c) the initial concentrations (10–50 mg/L).

pHs (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 are consistent with previous findings [35,36].

Based on Figure 4, the pH values of the aqueous solutions after completing the absorption process at all concentrations gradually shift to the basal state with increase in the contact period, accompanied by an increase in the absorption capacity of nickle(II) on γ-Fe_2O_3: PAN nanofibers. The large capacities of nickel(II) are 63.06, 136.26, and 154.16 mg/g in a period of 120 min (as seen in Figure 4(a)) and aqueous solution pHs 7.63, 7.2, and 6.21 (as seen in Figure 4(b)) when the initial concentration of nickel(II) increases in the aqueous solution from 10 mg/L to 50 mg/L (as seen in Figure 4(c)).

The behavior of adsorption capacity as a function of initial nickel(II) concentration appears to be consistent with the findings achieved in [37]. It is worth noting that PH∼8 has a high absorption capacity [12,14,38,39]. Table 1 summarizes the comparison of

the absorption capacity and the initial concentrations of nickel(II) for this research with the results of the absorption capacity of the composite nanofibers based on the initial concentrations of nickel(II) in aqueous solutions given in the previous research. Previous findings indicate that increase in the initial concentrations of nickel(II) leads to an increase in the absorption capacity of these ions on the surface of nanofibers. The absorption capacity depends on the efficiency of the functional groups of the filler materials, as well as the decrease in the diameter of the nanofibers, thus enhancing the rise of its surface area. Therefore, the superior performance of composite nanofibers is evident in the adsorption of nickel ions from aqueous solutions with high concentrations in the acidic state more than in the basic state. The competitive adsorption between available H^+ and metal ions hinders the adsorption, but the number of free nickel(II) is large, thus increasing the adsorption capacity of nickel(II) on the surface of nanofibers. This result is consistent with the previous findings [40,41].

The electrical conductivity of solutions was defined by the solution 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 reduced when increasing the contact time $0-120$ min. (Figure 5(a)). The minimum electrical conductivity rates are (4.15, 9.167, 17.73 mS/cm). In addition, the electrical conductivity is reduced upon increasing solution pH; then, the minimum value of electrical conductivity at pHs 7.63, 7.2, and 6.21 (as seen in Figure 5(b)) is determined when increasing the initial nickel(II) concentration in aqueous solution from 10 to 50 mg/L. Therefore, conductivity decreases because the number of ions per unit volume is reduced.

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

3.1.2. Mechanism of absorption

The adsorption mechanism verifies the performance of the composite nanofibers in removing nickel(II) from aqueous solutions prepared at concentrations (10–50 mg/L) and pH ($3.5-7.5$) for contact periods with aqueous solutions (0–120 min). Strengthening the polyacrylonitrile matrix with iron (γ-Fe_2O_3) nanoparticles expands the surface area of the composite nanofibers, reduces the diameter of the nanofibers, and increases magnetite. Moreover, the mechanism and kinetics of the adsorption process depend on several factors such as surface morphology and magnetic behavior of adsorption in the pseudo-first-order model [36,46].

The Lagergren rate (pseudo-first-order) is given in Eq. (5) as follows:

$$\frac{dq}{dt} = K_1(qe - qt), \tag{5}$$

where K_1 is the rate constant of pseudo-first-order adsorption (min^{-1}), q the amount of metal ion adsorbed at different times (mg/g), and qe the amount of metal ion adsorbed onto adsorbent in equilibrium (mg/g). After integrating with the initial conditions, Eq. (5) converts into Eq. (6) [33,47]:

$$\ln(qe - qt) = \ln qe - K_1 t$$

$$\text{Pseudo} - \text{first} - \text{order (Linear form)}. \tag{6}$$

All information about the absorption of nickel(II) ions by nanofibers was fitted into the pseudo-second-order adsorption kinetics according to Eq. (7) [11,48]:

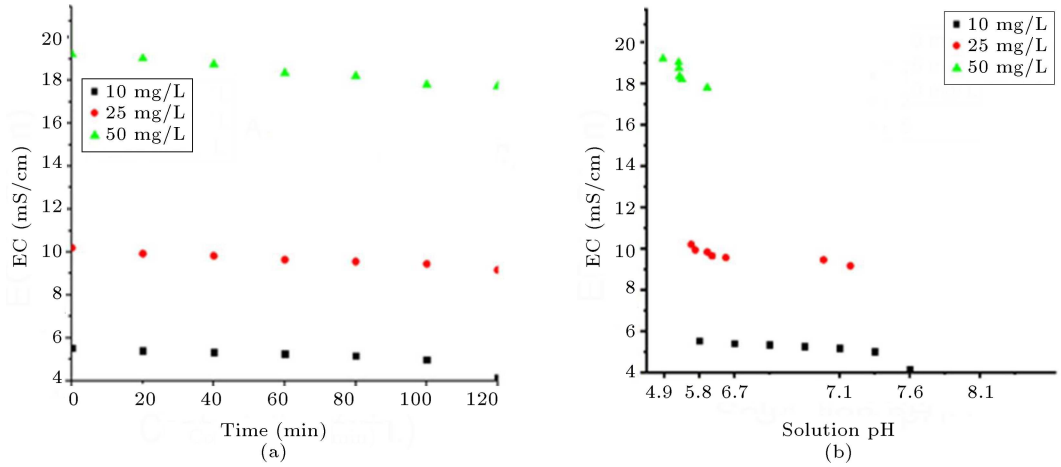

Figure 5. Electrical conductivity (mS/cm) before and after adsorption of nickel(II) from the aqueous solution by composite nanofibers (a) for many contact times (min) and (b) different solution pHs, with the initial concentration (10–50 mg/L).

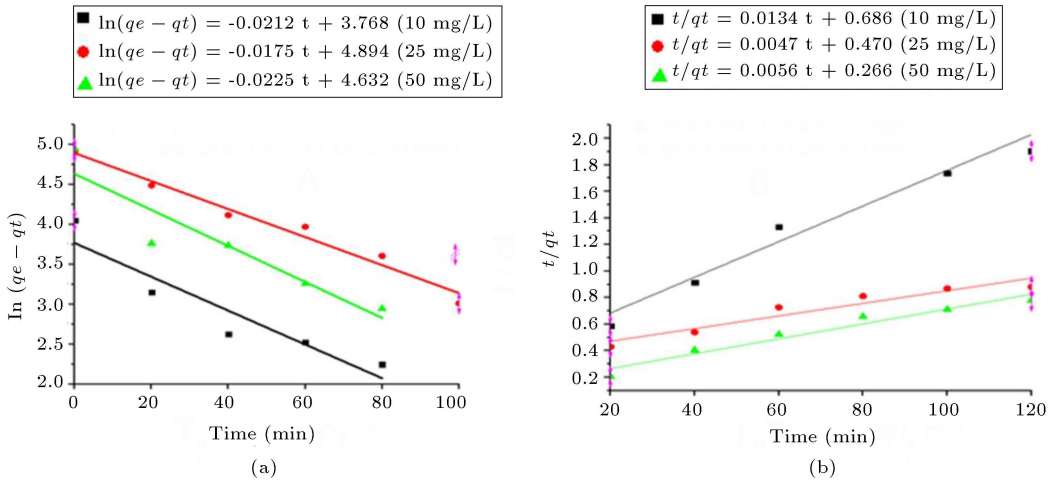

Figure 6. Adsorption kinetics of nickel(II) on composite γ-Fe_2O_3-PAN nanofibers by using (a) the pseudo first-order model represented by the relation between $\ln(qe - qt)$ and t, (b) Pseudo-second-order model represented by relation between t/qt and t, when increasing initial concentrations of aqueous solution (10–50 mg/L).

$$\frac{dqt}{dt} = K(qe - qt)^2. \tag{7}$$

After integrating Eq. (7) and rearranging it, Eq. (8) is achieved [12,18,20]:

$$\frac{t}{qt} = \frac{1}{K_2 qe^2} + \frac{1}{qe}t,$$

$$\text{Pseudo} - \text{second} - \text{order (Linear form)}, \tag{8}$$

where K_2 represents the second-order absorption rate constant (g/mg min), and $1/K_2qe^2$ represents the 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 6(a) and (b) when using nanofiber composites based on the absorption information of nickel ions referred to in Table 2. The table shows the comparison of the results of the adsorption kinetic parameters for Ni(II) by pseudo-first-order and pseudo-second-order models in previous and current research studies. The results of previous studies on the adsorption mechanism of nickel ions on the surface of the PAN: γ-Fe_2O_3 nanofibers with different initial concentrations and different polymeric nanofibers were used as adsorption materials. The correction coefficient increases in value with an increase in the initial concentration; the results of this research agree with those of all previous findings, especially in the case of polyacrylonitrile.

To investigate the sorption mechanism of nicle(II) on the surface of PAN: γ-Fe_2O_3 nanofibers, we estimated the maximum sorption capacity (q_{max}) for the pseudo-first-order model (Figure 6(a)). The q_{max} represents the interception of the relationship between the $\ln(qe - qt)$ and t (Eq. (6)), while q_{max} represents the (1/ slope) of the relation between t/qt and t(Eq. (8)) for the pseudo-second-order model (Figure 6(b)).

The increment rates in the maximum sorption capacity (q_{max}) of nickel(II) on the surface of PAN: γ-Fe_2O_3 nanofibers were 137.4% and 139.3% for the pseudo-first-order and pseudo-second-order models, respectively. Although the rate constant of sorption K_1 and K_2 increased slightly when increasing the initial concentration of nickel(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, better results were obtained using the pseudo-second-order model [49].

3.2. Physicochemical analysis of the wastewater

Sewage and groundwater pollutants are among the most important determinants for the use of these water sources for watering plants or benefiting from them in other fields. Among the important pollutants is TSS, which is an important indicator of the presence of Total Dissolved Solids (TDS) in water, and it consists of organic and inorganic materials that come from mining, crushing, washing materials, etc. They can be removed using micro-membranes and nano-membranes [50]. Reduction in the efficiency (%) of pollutant removal from wastewater can be determined via Eq. (3). Table 3 shows the results of spectrometer (Hach DR2800) for the three models applied to the limited concentrations of untreated sewage pollutants of water taken from (Al-Muaymira) site, as well as the limits of wastewater parameters according international standards. From the above table, we notice that the limits of pollutants have low concentration levels. In this work, the PAN: γ-Fe_2O_3 nanofibers were used to reduce these concentrations. Table 4 and Figure 7(a) show a significant reduction in the concentrations of these

Table 3. Physicochemical analysis of the Al-Muaymira wastewater in 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
(COD)	150–154 (mg/L)	1100	[67]
		120	[68]
(TSS)	129–135 (mg/L)	150	[56]
		800	[67]
(TP)	2.9–3.4 (mg/L)	25	[67]
		10	[69]
		5	[68]
(NO_3^{-2})	0.7–0.8 (mg/L)	3	[70]
		50	[68]
(NO_2^{-2})	0.567–0.587 (mg/L)	3	[70]
(CN)	0.045–0.09 (mg/L)	0.2	[67]
		2	[69]
(TN)	3.78–4.5 (mg/L)	20	[70]
		100	[67]
Collection of bacteria	3000-3600 (CFU/ ml)	300 CFU/ mL	[71]
		650 CFU/ mL	[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. physicochemical analysis of the Al-Muaymira wastewater in Iraq by using the spectrometer (Hach DR2800) after adsorption for many time periods with the presented composite nanofiber film.

	Pollutant concentrations (mg/L) at many absorption times				
Parameters	**Initial time**	**2 h**	**4 h**	**6 h**	**8 h**
(COD)	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_3^{-2})	0.7±06	1.4±0.055	1.4±0.06	1.4±0.04	1.4±0.038
Nitrite Ion (NO_2^{-2})	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 and cfu/ml calculation, EC: Electrical Conductivity.

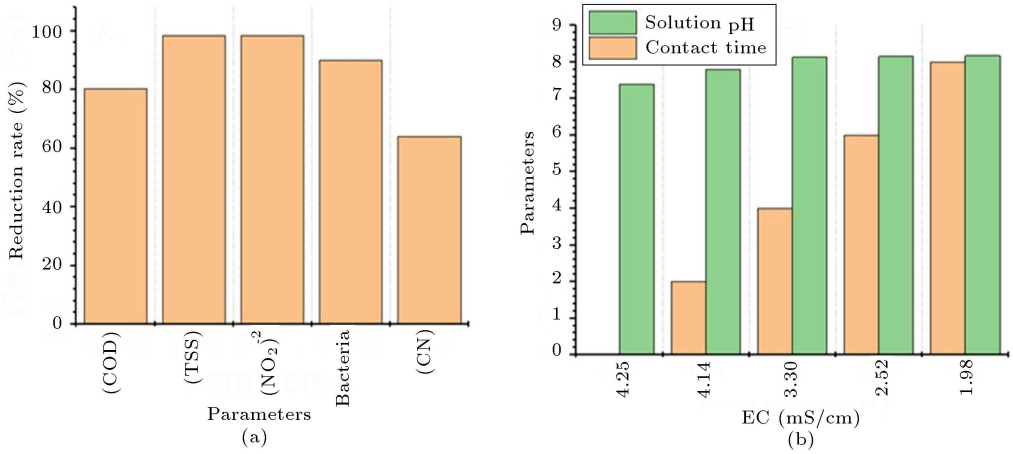

Figure 7. (a) Reduction percentages for the Al-Muaymira wastewater parameters in Iraq after eight hours of treatment with the presented composite nano-fiber film as well as such parameters as solution pH and contact time as a function of electrical conductivity of wastewater after treatment by γ-Fe_2O_3-PAN nanofibers.

pollutants in wastewater, and the reduction efficiency for pollutants reached 98.5, 80.26, 98.3, and 64% for TSS, COD, NO_2^{-2}, and CN, respectively, at time 8 h.

COD reduction (%) in this work was greater than the rate achieved in the case of using Ag-Clinoptilolite/Polyethersulfone nanofiber as an absorber material [51]. On the other hand, reduction in TSS removal efficiency (%) in this work was greater than that when using (PAN: Ag) or (PVDF: Ag) nanofibers [52]. In addition, CN is considered a toxic pollutant. Removing these pollutants implies removing bacteria even if they are within the minimum limits. The significant reduction of removal efficiency for CN and COD agreed with previous findings [53].

Pollutants such as nitrate (NO_3^{-2}), TP, and TN are all exposed to organic pollutants in wastewater. Because the studied samples have low concentrations of pollutants (TSS and COD), the removal efficiency (%) is approximately stable with increase in the nanofiber treatment period for some pollutants such as TP, while the role of nanofibers is negative in the reduction of nitrate (NO_3^{-2}) removal efficiency.

Because of some physical and chemical techniques such as absorption, ion exchange or reverse osmosis is employed to transfer pollution instead of removing it. This event occurred in increasing nitrate (NO_3^{-2}) concentrations after the nanofiber treatment process [54]. The treated wastewater concentrations by composite nanofibers at time 8 h for TSS and COD were 2 ± 0.94 and 34 ± 2.52 (mg/L), while the permissible limits for wastewater stipulated by the World Health Organization (WHO) were 600 and 250 (mg/L) [55]. In addition, the permissible wastewater limits 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 is enhanced with increase in the density or concentration of metal particles or ions having electrical conductivity. Figure 7(b) 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 increase in treatment time. This is evidence of the efficiency of nanofibers in removing pollutants by reducing the concentrations of these pollutants with an increase in the treatment time and wastewater pH. These results are consistent with the referenced study that used polymers as adsorbents to remove pollutants from wastewater [45].

3.3. Reduction of bacteria

The researchers have employed 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, plays a significant role in eliminating bacteria because it increases their efficiency by increasing the total surface area of exposure [58]. The bacterial log reduction is calculated using Eq. (9):

$$R(\%) = (A - B) * 100, \tag{9}$$

where R is the reduction in CFU, A the number of bacteria isolated from the inoculated electrospun nanofibers after defined time contact time, and B the number of bacteria isolated from the inoculated electrospun nanofibers at zero contact time [24]. The ratio between TN and 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 a higher bacterial concentration in sewage water. This result indicates an increase in TSS in the water

sewage. Table 4, Figure 7(a), and Figure 2(a) illustrate, similar to the above table, that the ratio (TN:TP) increases with an increase in the treatment periods by nanofibers, because bacteria cannot resist high alkaline conditions due to the high pH of the treated water upon increasing treatment periods [27]. Therefore, we notice a decrease in the total bacteria colony count from 3000 to 400 CFU/mL at time 8 h of the treatment (as seen in Figure 2(a)), accompanied by a significant rise in the efficiency (%) to reduce TSS. These results were in agreement with previous findings [59]. To compare the percentage of bacterial reduction in wastewater with that of coliform bacteria reduction to 1340 CFU/ml, which represents drinking water quality by nanofibers [60], we note that the rate of bacterial reduction by the utilized nanofibers reached 300 CFU/ml at time 8 h.

4. Conclusion

This study explained the efficiency of PAN: γ-Fe_2O_3 nanofibers in absorbing nickel(II) from aqueous solutions at low initial concentrations (10–50 mg/L) and reducing the concentrations of pollutants such as Total Suspended Solid (TSS), Chemical Oxygen Demand (COD), Total Phosphor (TP), Total Nitrogen (TN), and Cyanide (CN) and bacteria from real wastewater. Polyacrilonitrile (PAN): γ-Fe_2O_3 nanofibers had electromagnetic properties and a high surface area, which increased the absorption efficiency. The results of the adsorption kinetic parameters for nickel(II) by pseudo-first-order and pseudo-second-order models indicated that the maximum adsorption capacity of nickel(II) on the surface of PAN: γ-Fe_2O_3 nanofibers increased with increase in contact time, solution pH, and initial nickel(II) concentration. In addition, increasing the initial absorption rate in K_1 (min^{-1}) and K_2 (g mg^{-1} min^{-1}) was dependent on increasing the initial concentrations and the adsorbent materials used. In this work and previous studies, the composite nanofibers had a lower adsorption rate than other polymeric materials. Moreover, physico-chemical analysis of the Al-Muaymira wastewater in Iraq using the spectrometer (Hach DR2800) proved that the composite nanofibers had a high ability to reduce the pollutants such as TSS, COD, $(NO_2)^{-2}$, and CN from wastewater. The increase in the TN:TP ratio and contact time leads to the enhancement of the nanofiber efficiency in reducing bacteria in the wastewater, followed 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 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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Biographies

Salih Abbas Habeeb is an Assistant Professor at the Polymers and Petrochemical Industries Engineering Department, Materials College, University of Babylon, Al-Hilla, IRAQ. He received his BSc degree with distinction in Chemical Engineering from University of Technology, Iraq, MSc degree in Materials Engineering-Polymers Technology from University of Babylon, and PhD degree in in Polymer Nanotechnology from Razi University, Iran. He has authorized more than 20 international publications besides 10 publications in Scopus and web of science index. His interest is mainly in electrospun polymeric nano-fiber technology, especially with the physicochemical processes of water treatment by the incorporation of membranes. In addition to the composite bio-nanofibers for the drug delivery system, his projects have primarily dealt with membranes, oxidation-reduction, and catalysis processes for low-level pollutant removal. In addition, he has interests in polymer nano-composites such as rubber composite. Moreover, he teaches heat transfer, mass transfer, and process control for undergraduate students as well as the polymer nano-composite with advanced rubber technology for postgraduate studies at the Department of Polymers and Petrochemical Industries.

Baseem Ali Nadhim holds a BSc degree in Materials Engineering in 2008 and a MSc degree from Babylon University, College of Materials Engineering in Polymer and Petrochemical Industries in 2021. He is currently working at the Nanotechnology Laboratories at the Faculty of Materials Engineering.