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Synthesis, and Characterization of Magnetic Nano-particles using Opuntia Extract, and its Applications in Wastewater Treatment

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Abstract

Opuntia peels have several properties that can be used as an environmentally friendly and renewable synthesis of magnetic nanoparticles using the green synthesis method. The purpose of this study is to identify the properties of nanoparticles prepared from opuntiapeel extract and used in wastewater treatment. In this study, the efficiency of slurry methods such as coagulation, flocculation, and adsorption were compared using magnetic iron nanoparticles (O-FeNPs) with opuntia peels. The prepared iron nanoparticles were investigated and the surface properties of the particles were determined using X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier transform infrared (FT-IR).Several factors were applied to determine the efficiency of different treatment processes, such as contact time (0 to 60 minutes) and doses (0.1-1.0 g). The chemical oxygen demand (COD), biological oxygen demand (BOD_5), total kjelidahl nitrogen (TKN), total nitrogen (TN), ammonia (NH_3^+), phosphate (PO_4^{3-}), total suspended solid (TSS), and nitrate (NO_3^-), were measured for the wastewater before and after treatment. By examining the iron nanoparticles by FT-IR spectroscopy, the presence of biologically active opuntia particles on the surfaces of the nanoparticles was revealed. Also, the XRD pattern revealed that the nanoparticles are present in the oxide form as a phase of Fe_2O_3.The results shown among the treatment methods, flocculation and coagulation have lower efficiencies, while O-Fe NPS has higher efficiencies in removing pollutants from wastewater treatment. It was concluded that the O-Fe NPs removal efficiencies were 87.4%, 87.3%. 96.28%, 98.19%, 96.28% 34.4%, 86.4%, and 100% for COD, BOD_5, TKN, TN, NH_3^+, PO_4^{3-}, TSS, and NO_3^-, respectively. Itconfirmed that the magnetic nanoparticle/opuntiacomposite is an environmentally friendly adsorbent that can be effectively used in environmental applications such as wastewater treatment.

Keywords:wastewater treatments, coagulation, flocculation, opuntia, iron magnetic nanoparticles

1. Introduction

Water is one of the most important causes of life in humanity, so scientists and countries have made a lot of efforts to meet the needs of their countries for usable water, and due to the scarcity and lack of water to meet the needs of many countries[1], it was necessary to provide alternative methods for the existence of water sources, and the most important of these sources is water treatment for drainage.Wastewater contains organic and inorganic pollutants, suspended solids (TSS)[2]. Several studies have been conducted to reuse the treated water after the process of removing pollutants in various ways[3], such as adsorption processes, coagulants[4][5], ion

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separation[6], nanoparticles[7][7][8][9][10], wetland system [11][12], activated carbon[13][14], agro-waste materials[15], sedimentation, filtration [2], as well as biomaterials such as chicken bone ash[16][17].

One of these methods is the use of coagulants and flocculation, in which materials such as alum, ferric chloride, lime and polymers are used. These materials improve suspended particles in the water to settle, which helps in the treatment process and may have bad effects on environmental and health life[2][18][19][20][21]. The peels of plants and fruits are used as a natural coagulant in water treatment, such as fruit peels and moringa[22], and opuntia[2][23]. The other method that is used in the treatment process is the use of adsorbents because of their many advantages such as low cost, availability, environmental friendliness, and high efficiency, and sorbents that use orange peels, banana peels, opuntia, pomegranates, and potatoes [14][24][25][26][27][28]. The most modern and most efficient method at the present time is the use of nanoparticles, and they are prepared in an environmentally friendly manner without any chemicals, and at a low cost by means of extracting natural materials, the so-called green synthesis[29][17]. The natural product extracts are characterized by the presence of phenolic acids, which are antioxidants because they contain carboxyl and hydroxyl groups that are able to bind minerals. The active hydrogen may be responsible for the reduction of metal ions to metal nanoparticles[30][31][32][33].

The aim of this study is the use of different treatment methods utilizing opuntia extract for wastewater treatment. Such as adsorption method, coagulation/flocculation, and the use of opuntia with the help of iron nanoparticles by green synthesis.The three methods studied in this work were compared using the dose effect. In addition, Fourier transform infrared (FT-IR), scanning electron microscope(SEM) and x-ray diffraction(XRD) characterization of the prepared iron nanoparticles were also done.

2. Materials and methods

2.1 Preparation of coagulants, iron nanoparticles, and adsorbent

Opuntia fruits were collected from the Egyptian market. Impurities were removed from the opuntia peels, which were then cut into small pieces (1-2) cm (manually), the small pieces of opuntiawashed several times with tap water and then washed with distilled water. 200 grams of opuntia particles were introduced into distilled water. The extract solution was placed on a hot plate to boil for 2 hours. After this period, the extract formed was filtered through No. 1 filter paper, and then the extracts were stored at 4 °C in a refrigerator[6][34].

Opuntia magnetic iron nanoparticles were produced from opuntia extract, which was prepared by cutting the peels into small pieces and adding 5.0 g of these pieces to distilled water for 120 min at 95 °C, and the extract was filtered through filter paper No. 1, add 5 mlfrom the extract to the $FeCl_3/FeCl_2$ solution.A mixture of $FeCl_3.6H_2O$ and $FeCl_2.4H_2O$ was mixed with the extract solution, and the pH was adjusted to an alkaline medium by adding NaOH solution (drop by drop).At a temperature of 70 °C while mixing at 1000 rpm for 2 hours, the color of the reaction mixture changes to black, indicating the formation of iron nanoparticles[34][35]. Opuntia powder produced by opuntia peels was cut into small pieces, rinsed thoroughly with tap water, dried at 100-105 °C 24 h to remove water content, and sieved (60-90 µm) to obtain a more homogeneous particle size[36]. As shown inFigure 1, all methods were prepared and ready to use.

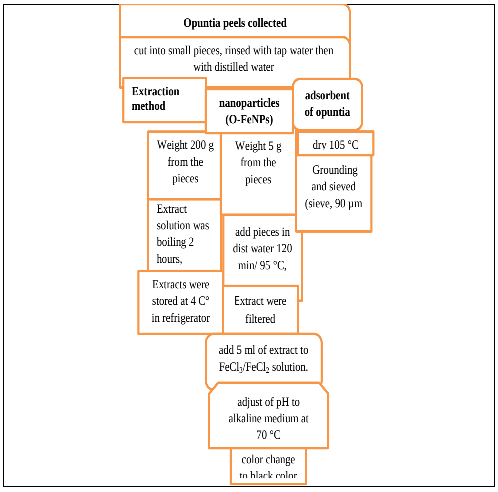

Figure 1: The adsorbents opuntia powder, O-Fe NPs, and opuntia extract solution are depicted schematically.

2.2 Characterization of opuntia an adsorbent, and iron nanoparticles

A scanning electron microscope (SEM) was used to study the surface morphology of opuntia adsorbents and O-Fe NPs. To investigate the structure of O-FeNPs, X-ray diffraction (XRD) was performed using a model (X'Pert Pro PAN analytical manufactured by PAN analytical B.V. Co., Netherlands) (ISO 9001/14001 KEMA - 0.75160). Fourier transform infrared (FT-IR) spectra were collected for adsorbents, and iron nanoparticles (ANE) using Cary, South San Francisco, and the United States of America, to study the functional group of opuntia adsorbents and O-Fe NPs.

2.3 Characterization of raw wastewater

Raw wastewater used for this study was drawn from the WWTP is located in New Cairo. These samples were collected over 24 hours and mixed for homogenised samples, measure various parameters for wastewatersuch as chemical oxygen demand (COD), biological oxygen demand (BOD_5), total kjeldahl nitrogen (TKN), total nitrogen (TN), ammonia (NH_3^+), phosphate (PO_4^{3-}), Sulfides, total suspended solid (TSS), and nitrate (NO_3^-), were measured for the wastewater before and after treatment. These wastewaters must be suitably treated to avoid environmental problems. The sample shows those mean values for 7.52, 1780, 3200, 582, 355, 362, 9.6, 19.8, 4.52, 0.723, and 10.2 mg/L, respectively. Table 1 shows the physicochemical composition of wastewater samples determined using the method described in APHA][37]

Table (1): Physicochemical characteristics of raw wastewater

No.	Test	Unit	Min.	Max.	*Aver.	**Limit[38]
1	pH	----	7.5	7.7	7.52	6.00 - 9.00
2	TDS	mg/L	618	680	1780	2000
3	EC	µs/cm	1112	1224	3200	250-750
4	Turbidity	NTU	122	144	133	---
5	TSS	mg/L	380	400	362	40
6	COD	mg/L	658	680	582	80
7	BOD_5	mg/L	394	408	355	40
8	PO_4^{3-}	mg/L	3.1	3.89	0.723	2.0
10	NH_3^+	mg/L	3.4	4.1	4.52	2.5
11	NO_3^-	mg/L	8.9	11.8	10.2	40
12	TKN	mg/L	7.276	8.774	9.6	15
13	TN	mg/L	16.176	20.574	19.8	----

Note: -NTU is Nephelometric Turbidity Unit,
*Average for 2 samples from wastewater.
* EEAA: Egyptian Environmental Association Affair, (law: 48/1982

2.4. Jar test experiments

The test was carried out using the different doses from three cases of treatment methods (opuntia adsorbents, coagulation/flocculation, and O-FeNPs) and was performed according to the protocol using the "Jar Test" in six beakers with a volume of 1 L. This apparatus is set to two stirring speeds: flush mixing at 300 rpm for one minute and flocculation at 40 rpm for thirty minutes. The sedimentation procedure takes 30 minutes. In addition, different doses of adsorbents (0.1, 0.5, and 1.0 g), extract solution (1 mL, 5 mL, and 10 mL), and O-Fe NPs (0.1. 0.5, and 1.0)g; then the supernatant was collected and subjected to the physico-chemical analyses. Finally, determine the optimum dose for all experiments. The removal efficiency (R%) was determined by Equation (1)[39].

$$R\% = \frac{Co-Ce}{Co} \times 100 \qquad (1)$$

Where R% is the removal efficiency, C_0 is the initial concentration (mg/L), and C_e is the concentration after adsorption (mg/L).

3. Results

3.1 Characteristic of adsorbents

3.1.1. Surface Topography

Figure (2) shows SEM micrographs of both O-Fe NPs and opuntia adsorbent, which appear to have a smooth surface, porous structures, and a variety of pore sizes, indicating that these surface properties will result in high pollutants binding due to the available binding with pollutants.

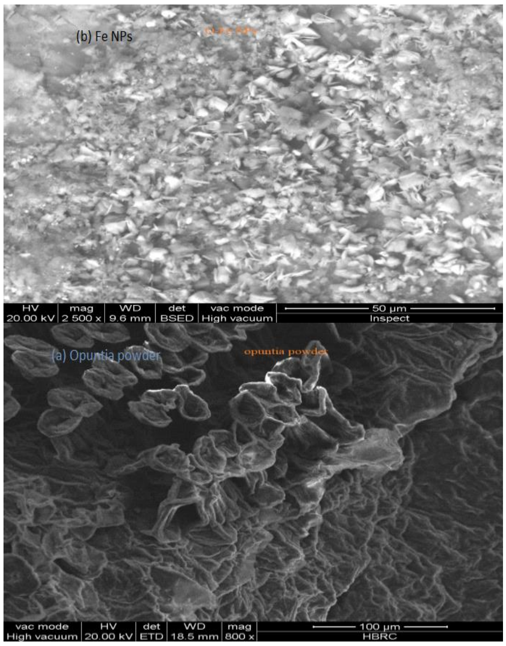

Figure 2: SEM of the surface of (a) an adsorbents powder, and (b) O-Fe NPs

3.1.2 FTIR analysis

FT-IR analysis was done to investigate the functional groups of adsorbents that are responsible for the adsorption shown in Figures (3, 4). The FT-IR spectrum of adsorbents was detected in the range of 1000–4000 cm^{-1}, there are peaks, with peaks and a strong band of amine or hydroxyl (N–H or –OH) groups at wavenumber 3400[40].The abundance of hydroxyl groups from cellulose is attributed to the pollutants' binding mechanism of adsorption, in which aqueous medium favours ion exchange or complexationwith contaminants[40]. The band presented at 2923 cm^{-1} may indicate the –C–H stretching vibration from aliphatic compounds. The absorption at 1598 cm^{-1} indicated N–H bending in the adsorbent. The C=N stretching in heterocyclic rings was also specified at wavenumber 1437 cm^{-1}. The peak at 1316 cm^{-1} because of the C–OH stretching vibration of alcohols. Finally, the carboxylic acids were observed at 1156 cm^{-1} of the adsorbents[26].

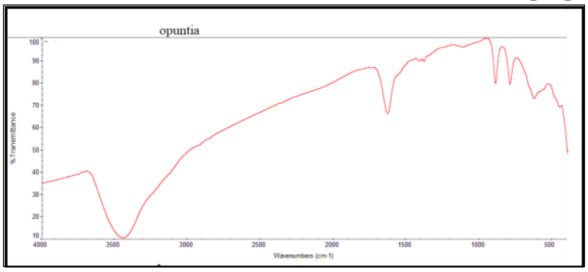

Figure 3: FT-IR spectrum of the opuntia powder

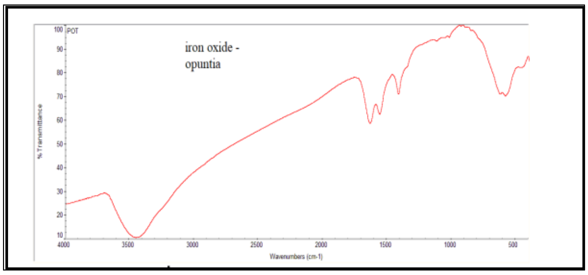

Figure 4: FT-IR spectrum of O-Fe NPs adsorbent

3.1.3 XRD analysis

The powder x-ray diffraction pattern of O-Fe NPs as an adsorbent is shown in Figure (5). The crystalline nature was reflected by the sharp and intense peaks at 1.8°, 3.0°, 3.5°, and 4.3° and the less intense peaks at 1.8°, 4.3°, and 3.0°. However, due to its amorphous nature, these peaks shifted and broadened towards lower values.

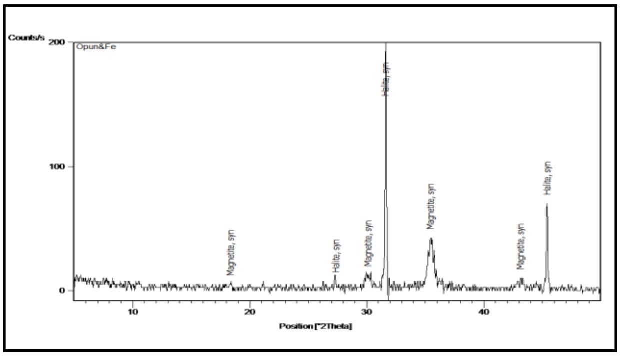

Figure 5: XRD spectrum of O-Fe NPs

3.2. Effect of doses

3.2.1 Effect of optimal volume for coagulants

Figures (6,7) show the variation in water quality as a function of the volume of the added bioflocculants. We evaluated the efficiency of opuntia for the removal of COD, BOD_5, TKN, TN, ammonia, phosphate, TSS, and nitrate from wastewater, as well as the effect of the water's pH and TDS on such efficiency. They assessed the effectiveness of opuntia as coagulants/flocculants by measuring COD, BOD_5, TKN, TN, ammonia, phosphate, TSS, and nitrate removal at various coagulant doses (1, 5, and 10 mL), When used at doses of 10 mL, they achieved removal percentages of 14.43, 14.92, 14.36, 28.35, 40.70, 40.41, 40.70, and 40.13 for COD, BOD_5, and TSS, respectively. Phosphate (TKN, TN, ammonia, and nitrate, respectively)[21][41][42]. Coagulant removal efficiency increases with increasing coagulant doses due to increased surface area availability at higher coagulant doses. The final pH, conductivity and TDS values are 8.36, 2570 $\mu S.cm^{-1}$, 1430 mg/L, respectively; the pH value is almost equal to the initial values. By comparing these results to those obtained by other bio-flocculants, we can conclude that this method has no effect on these parameters[43][18].

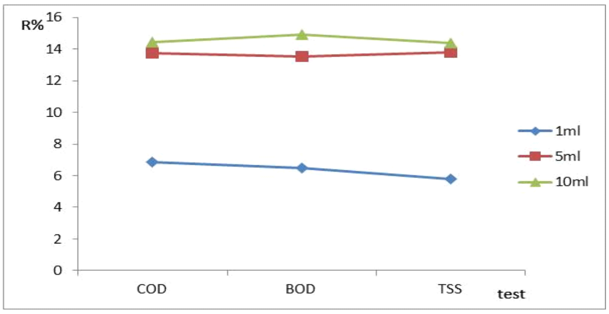

Figure (6): Removal of COD, TSS and BOD_5 of the treated wateraccording to the doses of coagulants added

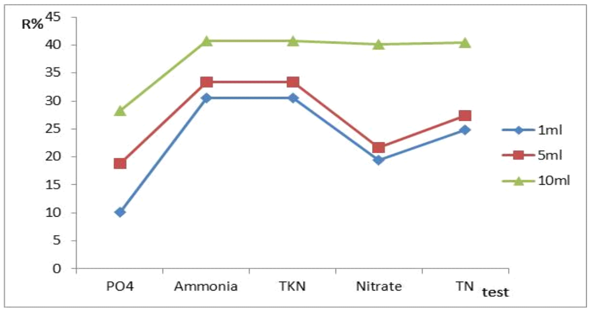

Figure (7): Removal (R%) of TKN, TN, Ammonia, Phosphate, and Nitrate of the treated water according to the doses of coagulants added

3.2.2 Effect of different doses from opuntia powder as adsorbent

The effect of bio-sorbent dosage on the biosorption of COD, BOD_5, TSS, TN, TKN, ammonia, nitrate, and phosphate were determined within the range of bio-sorbent dose 0.1–1.0 g/L, and the results are characterised in Figure (8). It is identified that the rise in bio-sorbent concentration caused an increase in the percent bio-sorption of COD, BOD_5, TSS, TN, TKN, ammonia, nitrate, and phosphate due to the increased availability of the surface area or transferable active sites but reduced the bio-sorption due to the incomplete accumulation or overlapping of bio-sorbent, which results in a reduction in active surface area for the bio-sorption [44]. Figure 9 shows that removal efficiency increased with increasing bio-sorbent dose for COD, BOD_5, TSS, TN, TKN, ammonia, nitrate, and phosphate, but not significantly after a definite dosage. The optimum amounts of opuntia as adsorbent dose for additional bio-sorption experiments were designated as 1.0 g/L for COD, BOD_5, TSS, TN, TKN, ammonia, nitrate The removal of the COD, BOD_5, TSS, TN, TKN, ammonia, nitrate, and phosphate is 58.59%, 59.43%, 58.56%, 81.46%, 94.12%, 94.12%, 69.48%, and 41.21%[2].

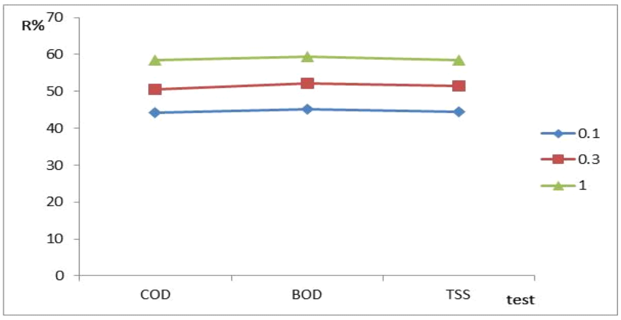

Figure (8): Removal of COD, TSS and BOD_5 of treated water according to the doses of adsorbent added

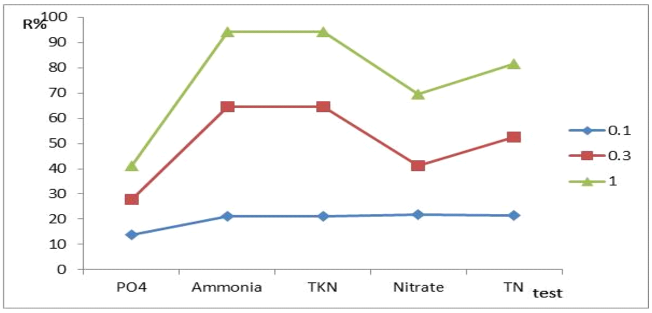

Figure (9): Removal (R%) of TKN, TN, Ammonia, Phosphate, and Nitrate of treated water according to the doses of adsorbent added

3.2.3 Effect of different adsorbent doses from O-Fe NPs as adsorbent

Iron nanomaterial adsorbents are important for removing COD, BOD_5, TSS, TN, TKN, ammonia, nitrate, and phosphate from wastewater.

Nano-iron oxides have been found to be promising adsorbents for removing COD, BOD_5, TSS, TN, TKN, ammonia, nitrate, and phosphate. Nano-oxide particles with certain characteristics (including a small volume, a large specific surface area, and strong magnetism) have been found to strongly adsorb COD, BOD_5, TSS, TN, TKN, ammonia, nitrate, and phosphate[45].

Iron nanoparticles O-FeNPs were used as an adsorbent to remove COD, BOD_5, TSS, TN, TKN, ammonia, nitrate, and phosphate from wastewater, with removal rates ranging from 87.32%, 86.46%, 34.43%, 98.19%, 96.28%, 87.57%, 100%, and 96.28%), as shown in Figures (10, 11).

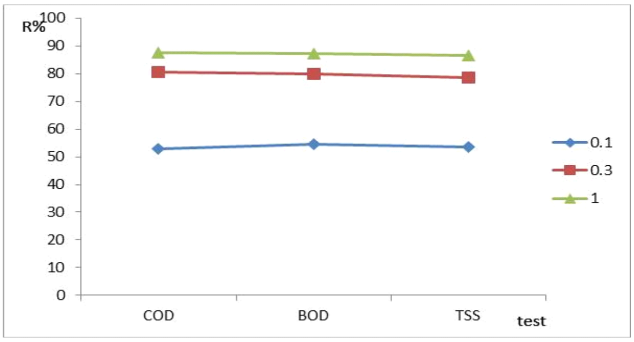

Figure (10): Removal of COD, TSS and BOD_5 of treated water according to the doses of O-Fe NPs added

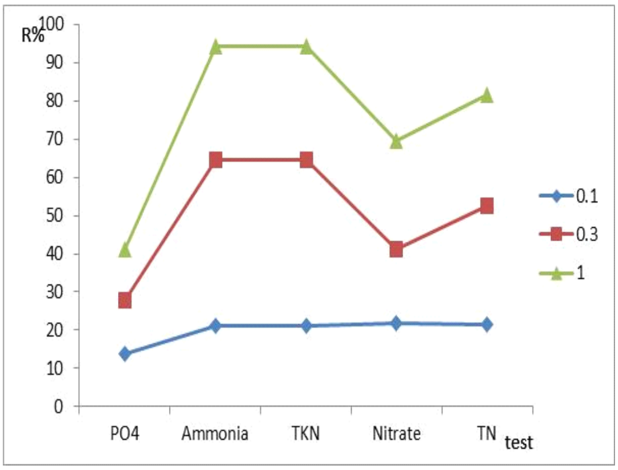

Figure (11): Removal of TKN, TN, Ammonia, Phosphate, and Nitrate of treated water according to the doses of O-FeNPs added

3.3 Comparison of three application of opuntia for wastewater treatment

Table (2) showed us the results of the comparison between forms of opuntia for the removal of pollutants from wastewater. From these results, it appeared that the opuntia extract had significantly lower efficiency than the adsorbent, and O-Fe NPs [2].

Table 2: The comparison of different treatment methods for removal of pollutants from wastewater

Test	Residual concentration of pollutants (mg/L)			Removal efficiency of opuntia (%)			* Limits [38]
	Coagulant 10ml/L	adsorbent 1.0g/L	O-Fe/NPs 1.0g/L	Coagulant 10ml/L	adsorbent 1.0g/L	O-Fe/NPs 1.0g/L	
COD	498	241	73	14.4	58.5	87.57	6.5-9.0
BOD_5	302	144	45	14.9	59.4	87.32	2000
TSS	310	150	49	14.3	58.5	86.46	---
PO_4^{3-}	0.51	0.42	0.4	28.3	41.2	34.43	80
NH_3^+	2.68	0.26	0.2	40.7	94.1	96.28	40
TKN	5.7	0.56	0.3	40.7	94.1	96.28	40
NO_3^-	6.12	3.12	0	40.1	69.4	100	2.0
TN	11.8	3.68	0.3	40.4	81.4	98.19	---

* EEAA: Egyptian Environmental Association Affair, Law Limits: 48/1982

4. Conclusion

The results presented in this work provide a clearer idea, technical potential, effectiveness, and environmental of an adsorbent, iron nanoparticles, and coagulation/flocculation process with wastewater, and the use of opuntia as different treatment methods to improve the wastewater quality, in terms of COD, BOD, TSS, TN, TKN, ammonia, nitrate, and phosphate. The coagulation-flocculation extraction recovers 1 L of extract by using 200 g raw material. The opuntia extract has a half-life of one month under storage conditions at 4 ° C. The best reduction rate is obtained when used at a dose of 10 mL, achieving removal percents of (14.43, 14.92, 14.36, 28.35, 40.70, 40.41, 40.70, and 40.13)% for COD, BOD, TSS, PO_4, TKN, TN, ammonia, and nitrate, respectively, no effect on TDS, conductivity (EC), and pH of the effluent sample. The treatment conducted by wastewater using O-Fe NPs had significantly better removal efficiencies for COD, BOD, TSS, TN, TKN, ammonia, nitrate, and phosphate (87.45, 87.32, 86.46, 96.28, 98.19, 34.4, 100, and 96.28) % in the treated water).

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