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Removal of PPD dye from its aqueous solution by Adsorption using the Cadmium oxide nanoparticles

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ABSTRACT— The manufacture of Cadmium oxide nanoparticles utilizing a green method is the subject of this research (environmentally friendly). Using the tomato leaf plant's aqueous extract. Using an adsorption batch technique, this oxide was employed to remove tincturepaea-Phenylenediamine from its aqueous solutions. To describe and study the surface, several techniques such as FT-IR, XRD, SEM, TEM, and AFM were applied. All of the reflection peaks with relative intensities of different planes, as determined by XRD analysis, indicate the existence of CdO, and the spectrum revealed that the particle size obtained was around (48.28 nm), which corresponded to those estimated by SEM and TEM. The sizes of the CdO particles were measured using SEM, TEM, and AFM, and they were found to be in the nanoscale range. The influence of contact time, adsorbent, and other variables the thermodynamic parameters for the influence of temperature were calculated after studying the dosage and beginning concentration. And the effect of the change in the acidity function, as well as the effect of increasing the temperature. Through the results, it was found that the best adsorption occurred at a time of 90 minutes and at a quantity of (0.2 g) of the adsorbent substance, as well as at the (PH = 5) the adsorption rate was at its best and at a temperature of (338 K). Recorded the best adsorption rate Among the results obtained when applying the Langmuir and Freundlich isotherms, the adsorption processes were of a physical nature.

KEYWORDS: Adsorption, Removal, Cadmium Oxide, Nanoparticle, Green chemistry.

1. INTRODUCTION

The importance of water for humans and other living organisms is well known, and no one overlooks the large space occupied by water from the globe, which makes up 71 percent of the earth and 70% of the human body, and studies have shown that the small cell relies on water in its construction. If the water is polluted, a large part of the body is polluted. This means that water contamination is the most hazardous sort of pollution at all levels [1]. Despite the necessity of water for life, whether for drinking, irrigation, or power generation, and its usage in a variety of businesses, humans pollute it and make it unfit for use by disposing of waste and pollutants near its sources [2]. Water pollution is defined as the occurrence of changes in the nature, quality, and properties of water that render it unfit for use, as a result of the addition of foreign or polluting substances such as chemicals, heavy metals, or toxic dyes, as dyes are one of the most dangerous pollutants that cause human diseases, and these dyes include para-phenylene diamine. We must proceed to tackle such problems that may pose a threat to human health, and scientists have been able to get rid of these contaminants in numerous ways, including chemical and electrical precipitation, extraction, leaching, evaporation, and adsorption methods, which were used in our study [3- 6]. Recent advances in nanotechnology have resulted in the development of adsorbed nanoparticles for separation applications, which take advantage of their morphology, small size, and large surface area. These materials are used in the removal technology, where the large surface area of nanoparticles provides many sites on the absorbent material, increasing the absorption capacity. In the advanced development of water treatment technologies, nanoparticles may offer unique new features that can be employed to extract heavy metals from water or waste water. Diverse varieties of green nanoparticles with well-defined chemical composition, size, and morphology have been synthesized using various methods in recent years, and their applications have been investigated in a variety of cutting-edge technical domains [7]. Other. Due to their energy efficiency, cheap cost, and non-toxic behavior of metal nanoparticle production technique, numerous extracts and plant products have garnered attention in recent years [8]. Aqueous extract of tomato leaves was utilized to make Nano cadmium oxide, which was then used as a reducing agent.

2. METHODS and MATERIALS

2.1 Adsorbate

By dissolving (0.5 g) of para-phenylenediamine (PPD) dye in deionized water, a standard solution of 1000 ppm para-phenylenediamine (PPD) dye was created. The standard solution was then made at various concentrations (10, 20, 30, 40, 50, 60, 70 ppm). (UV-VIS) technology was used to determine the absorbance of this solution at a wavelength (max) of 453 nm. Dye solution titration (PPD) at the longest wavelength of 453 nm, a series of standard solutions of various concentrations (5, 15, 25, and 35) was constructed using the Beer-Lambert equation between absorption and concentration and the obtained results. Standard calibration curve with a twist the dye solution's titration curve is depicted in figure (1).

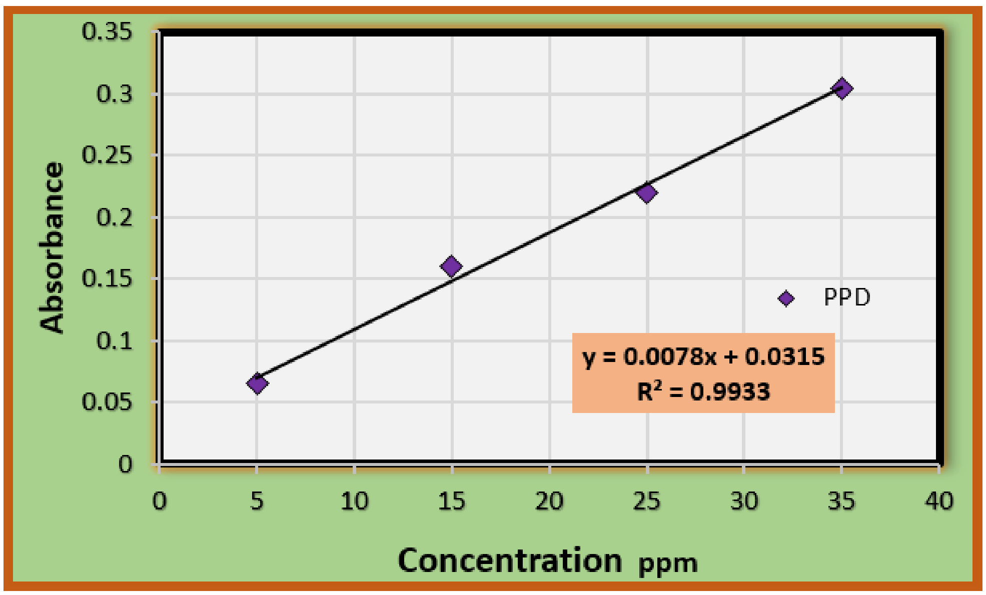

Figure: 1 represents the standard titration curve of PPD dye.

2.1.1 Preparation of the plant extract, not the tomato leaves

The plant extract of tomato leaves was prepared by collecting tomato leaves from one of the villages of Anbar Governorate. The leaves are dried away from the shadows, then grinded with an electric grinder to obtain a fine powder, then sieved with a sieve with a diameter of (212 nm) to get rid of the remaining impurities and plankton, Where the powder is stored in. Shade away from the light. (2.5 g) of the resulting powder was weighed into a (500 ml) glass beaker and (250 ml) deionized water was added. Taking into account the continuous stirring by magnetic stirrer The mixture was heated to a temperature (80 ° C) for one hour to obtain a brown extract, then the extract was cooled to room temperature and filtered by filter papers to obtain the solution, which we put in test tubes and centrifuges at 1200 rpm for a period of time. 8 minutes to get rid of the remaining fibers [9].

Nano- Cadmium oxide was prepared by the green method and using the previously prepared plant extract from tomato leaves, where (0.5 g) of Cadmium nitrate (Cd $(NO_3)_2$) was dissolved in (150 ml) of deionized water with continuous stirring. For 15 minutes at room temperature, then (15ml) of the plant extract was gradually added using the burette, where the color changed from white to light yellow, and then the temperature was gradually raised to (75°C), where the pH was adjusted by adding (0.1M)) of sodium hydroxide (NaOH) in the form of drops until the pH was equalized and the solution became neutral (PH = 7) Where the color changed to light brown, the solution was left to cool and then the filtrate was separated by a centrifuge, the filtrate was collected and washed with deionized water and absolute ethanol and dried at a temperature of (60°C) for three hours and the powder was kept at a low temperature [9]. Then the powder is calcined at a temperature of 400 °C for two hours by means of a ceramic lid and figure (2) prepared cadmium oxide powder.

Figure (2): Nano- cadmium oxide prepared by the green method

2.2 Adsorption study

Several volumetric flasks were prepared, each containing 50 ml (5 ml) of (PPD) solution at 1000ppm (45 ml) of distilled water (pH 5), and (0.05g) of the pre-prepared (CdO.NPs). At varied intervals of 15, 30, 45, 60, 75, 90, 105, and 120 minutes, the beaker was placed in a (water-bath mixing equipment) at 150 rpm and room temperature (298 K). The concentrations of the metal solution were obtained using atomic absorption and investigating the parameters that affect the adsorption of the two metals on (CdO NPs), including contact time, initial concentration, and amount of adsorbent. The percentage of time spent removing Using the calculation below [10], [11], the percentage of the Pigment removed (PPD) was computed.

$$R\ \% = \frac{Co - Ce * 100}{Co}$$

Where: (R %) is percentage removal of the metals, (C_o) is initial concentration (in binary system) of metals ions (mg/L), (C_e) is concentration of Lead ions after removal (mg/L).

2.2.1 Experiment of adsorption isotherm

Adsorption isotherm to remove tincture (PPD) from (CdO. NPs) at 25°C, pH (5), 0.05g adsorbent, 150 rpm stirring speed, and 90 minutes contact time on ready surface. The tincture concentration (PPD) solution (50 mL) was administered. The adsorption sample is: According to Complete Experiments (AAS), the adsorption sample is: The equation [10, 11] was used to calculate it.

$$Qe = \frac{(C_o - Ce) * v}{m}$$

Where :(Qe), the adsorption quantity of the surface nanoparticles at equilibrium (mg/g), (C_o) means the initial adsorbent concentration (mg/L). (Ce), equilibrium concentration of metals after adsorption has occurred (mg/L), (V), volume of aqueous solution (L), (m), weight of metal oxide nanoparticles (g).

3. RESULTS and DISCUSSION

3.1 XRD examination of CdO- NPs

The XRD spectra of CdO- NPs were studied in order to evaluate the crystalline phase and crystallite orientation. XRD pattern of is presented in Fig. (3). shows preferred orientation along (111) plane and other peaks are also observed with (200), (220), (311) and (222) planes. XRD pattern shows that CdO- NPs are polycrystalline in nature with cubic structure (JCPDS card no. 05-0640) [12], [13]. The crystallite size of the CdO NPs was obtained by well-known Scherer's formula [14], [15]. The crystallite size was calculated by resolving the highest intensity peak. In present study the estimated crystallite size for CdO-NPs was (48.28 nm).

$$D = \frac{0.9\lambda}{\beta cos\theta}$$

Where: (D) is the crystallite size, (λ) is the wavelength of radiation, (θ) is the Bragg's angle, (β) is the full width at half maximum (FWHM).

The found particle size of the (CdO.NP) is (48.28 nm). The presence of sharp peaks in XRD samples and particle size of less than 100 nm refers to the Nano- crystalline nature of the surface.

Table 1: The strongest three peaks in XRD of (CdO-NPs)

No	2θ (deg)	dǍ	FWHM (deg)	Intensity (counts)
1	**32.7692**	**2.73301**	**0.1968**	**90.14**
2	**38.1571**	**3.43270**	**0.2952**	**72.37**
3	**55.1758**	**1.74073**	**0.5904**	**15.76**

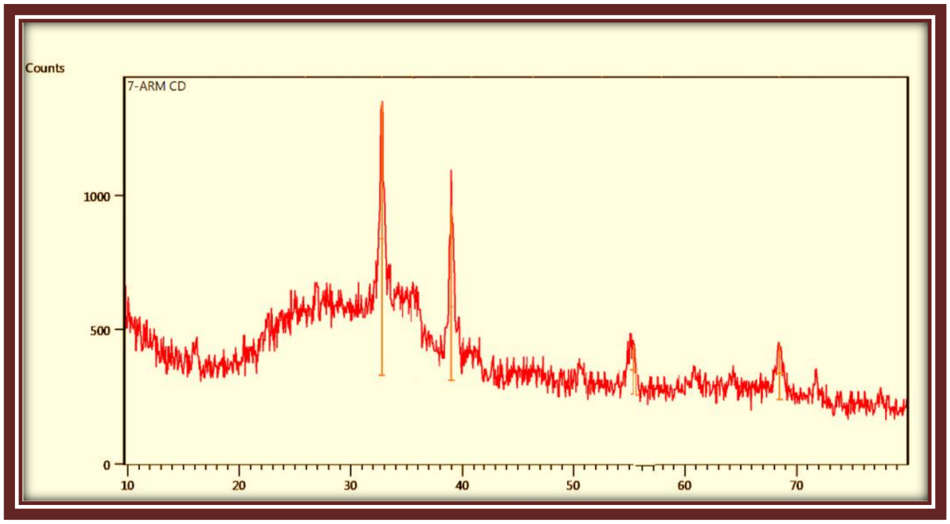

Figure 3: X–Ray Diffraction (XRD) profile of the CdO NPs synthesized nanoparticles by tomato leaf extract.

3.2 FT-IR analysis of CdO NPs

FTIR (Fourier Transform Infrared Spectroscopy) is a useful method for determining the functional groups of a substance. The FTIR spectrum was used to analyze the modes of vibration of chemical bonds in CdO Nano powder, and functional groups were recorded in the 400e4000 cm1 region. After filtration and centrifugation, the FT-IR spectrum fig (4) indicates no residual organic molecules like NO3. The stretching CdO modes [16] are related to the less intense peaks at 567, 505, 470, and 447 cm-1. The occurrence of these four distinct CdO stretching is attributed to the metal-oxygen stretching of CdO modes confirms the Monteponite CdO nanoparticles' phase purity.

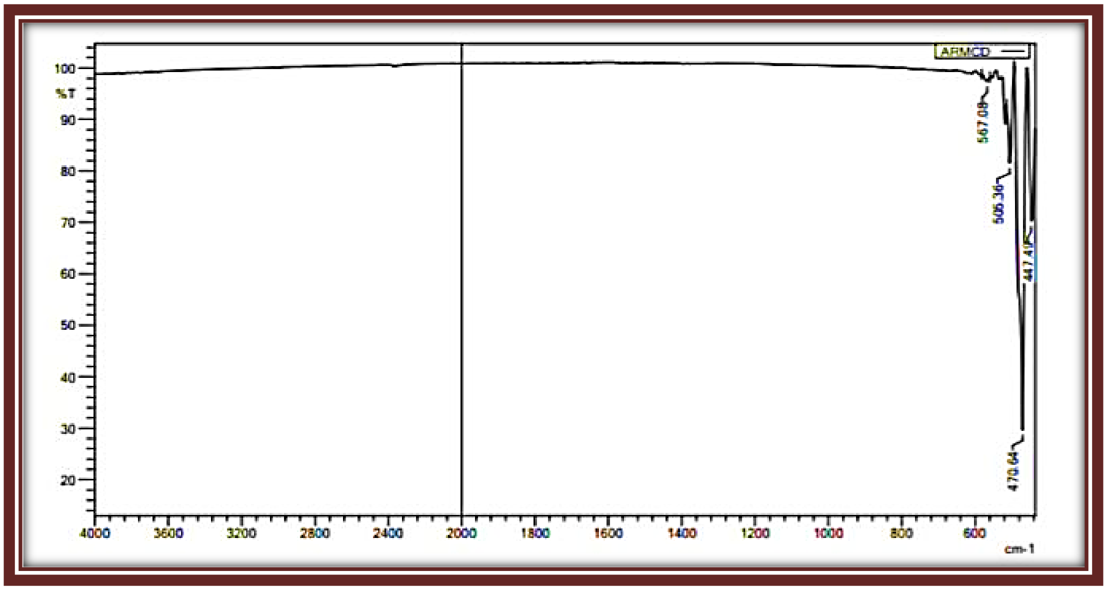

Figure 4: FT-IR analysis of CdO NPs synthesized by tomato leaf extracts (TLE CdO NPs)

3.3 Scanning electron Microscope

Figure 5 shows a High-Resolution Scanning Electron Microscopy (HRSEM) image of annealed CdO nanoparticles after 2 hours at 500°C [17]. Agglomerated clusters of varied sizes up to 80 nm in spatial extension make up the powdered sample. The clusters are formed by smaller agglomerated quasi-spherical nanoparticles with diameters as 33.67 nm, according to a more precise examination carried out within the limits of the HRSEM unit's resolution.

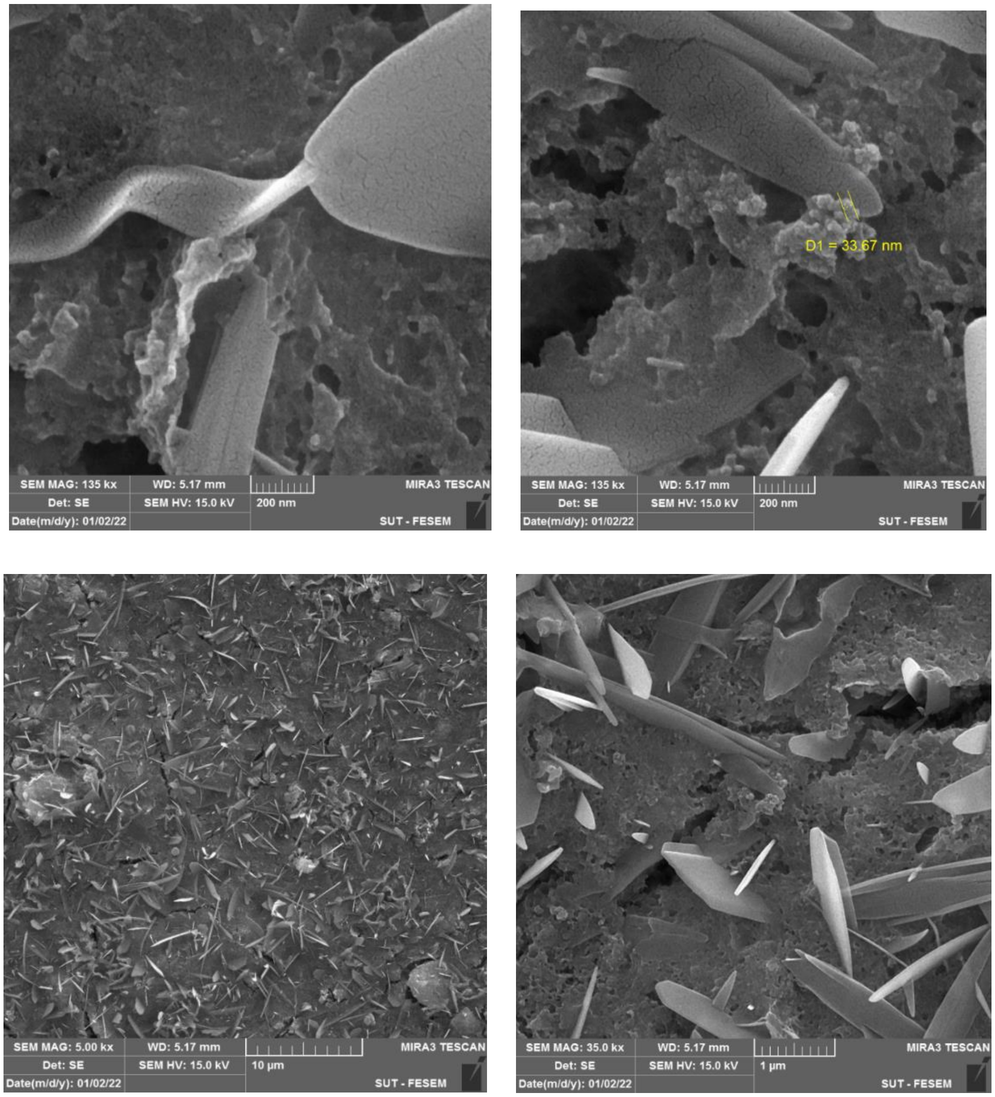

Figure (5): SEM images of cadmium (II) oxide nanoparticles prepare using tomato leaf extracts (TLE CdO NPs)).

3.4 Transmission electron microscope

Figure 6 shows a high-resolution transmission electron micrograph TEM! Of a CdO calcinated at 500 °C. The green synthesized CdO-NPs have a size range of 60–80 nm, according to TEM analysis [18].

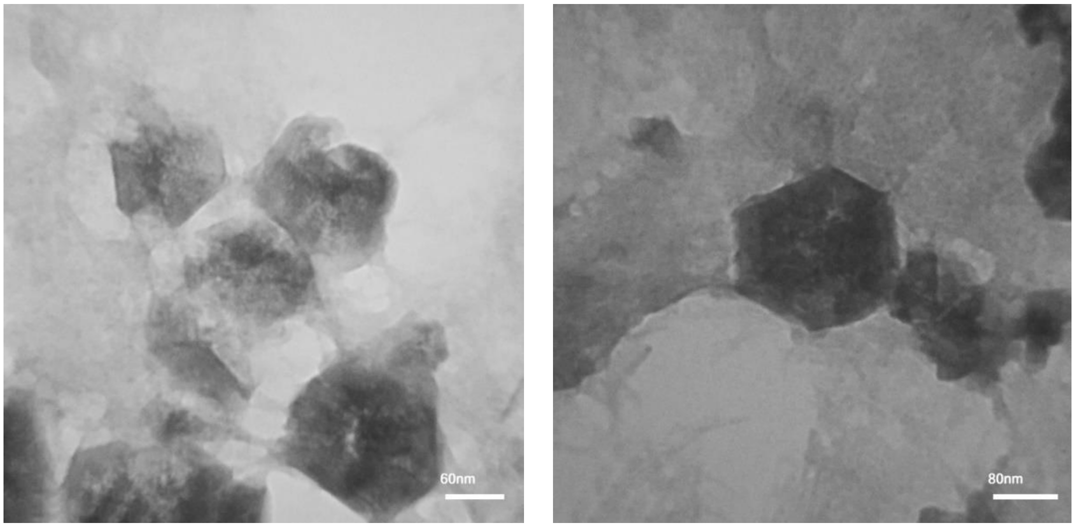

Fig. 6: TEM images of synthesized CdONPs

3.5 Atomic force microscope

The surface morphologies of CdO were studied using the atomic force microscopy (AFM) technique, as illustrated in Fig.8. The pure powder's The Average grain size, roughness, and root mean square are (52.9., 1.5, and 2.10), respectively [19].

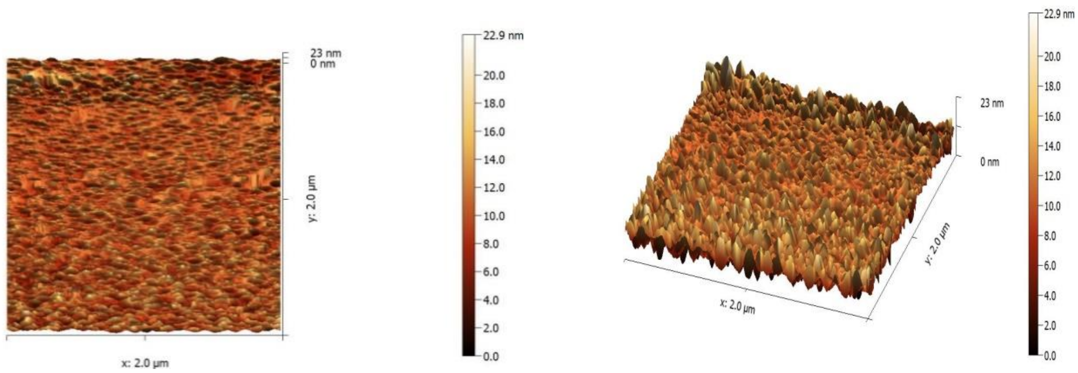

Fig. 7. The atomic force microscope 2-D and 3-D Image of prepared CdO-NPs

3.6 Factors affecting adsorption

3.6.1 The Effect of contact time on adsorption

The effect of the contact time on the removal of [tincture (PPD)] on the (CdO-NPs) was studied with contact time of (15, 30, 45, 60, 75, 90,105 and 120) min at 298 K, concentration 100 mg/L of each metal ions, and pH=5 Figure 8 explains the change in percentage removal with contact time. As we have seen, the equilibrium time required for pigment absorption (PPD) is approximately (105 min). The effect of contact time on the removal of dye using (CdO.NPs) shows that the increase in the percentage of removal at the beginning of the contact time with the dye (PPD), and then there was a gradual decrease in the percentage of removal of the dye, and the speed can be the initial increase in the rate followed at a slow rate in later stages Due to the availability of excess adsorption sites on the adsorbents [20]. The high initial absorption rate may be due to the ion exchange followed by a slow chemical reaction of the active groups in the sample [21], and the difficult to occupy the remaining vacant surface sites due to the strong repulsion. The dye solution used has to travel farther and deeper into the pores to encounter much greater resistance.

Figure (8): Effect of contact time of adsorption of tincture (PPD) on (CdO -NPs)

3.6.2 Effect of adsorbent quantity on adsorption

Effect of the amount of adsorbents on dye uptake (PPD). Where dye removal (PPD) was studied on (CdO-NPs.) prepared by the green method using tomato leaves and using different amounts of oxide (0.01, 0.05, 0.1, 0.15, 0.2) g at 298 K, fixed concentration of dye solution (100 mg / l), pH 5 with contact time was 105 min. Effect of the quantity of adsorbents on the removal of pigment (PPD) Figure (9) shows the percentage of removal with each quantity covered. The amount of cadmium oxide nanoparticles can be observed, and the removal of dye can be the largest part of the percentage of refresh removal in the place of adsorption, and the optimum amount of adsorption can be an idea when the dye is removed (PPD) is (0.1 g) and after 0.1 g, it increased Little clearance rate [22].

Figure (9): Effect of adsorbent quantity on adsorption

3.6.3 The effect of initial concentration

The adsorption of dye (PPD) from an aqueous solution to (CdO-NPs) was first studied under optimal conditions, using different initial concentrations of the aqueous solution (10, 20, 30, 40, 50, 60, 70 ppm) of the dye (PPD). The results in Figure (10) showed that the effect of the initial concentration indicates a slight decrease in the removal with increasing the initial concentration of the dye (PPD). On the surface (CdO-NPs). The small decrease in the percentage of removal at higher concentration can be attributed to the limited number of active sites for the cadmium oxide nanoparticle adsorbent, which becomes more saturated with increasing concentration of the dye used, and the removal of pigment (PPD). The (CdO-NPs) is high due to the small size of the nanoparticles and the high surface area which has a lot of active adsorption sites that will be available [23].

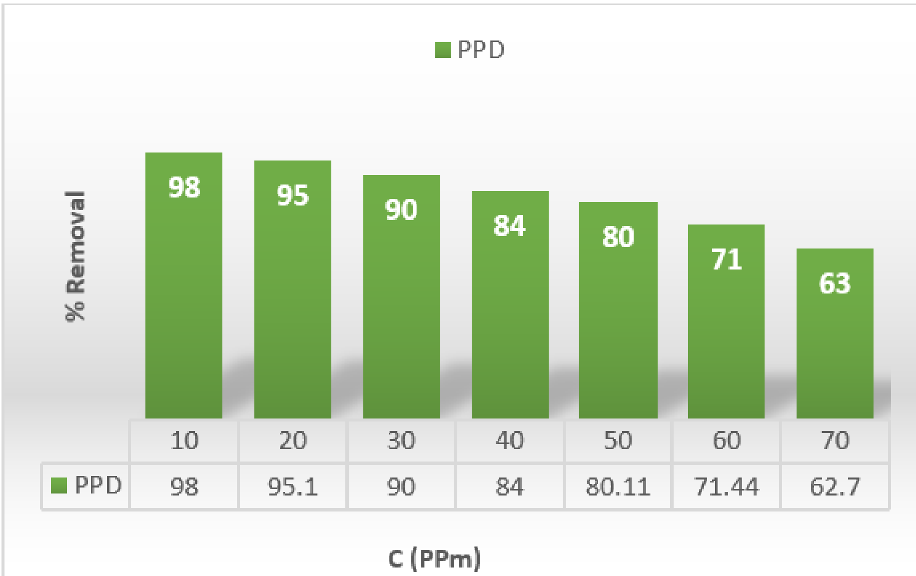

Figure (10): Effect of (initial concentration) on the adsorption of tincture (PPD). On (CdO-NPs)

3.6.4 Adsorption Effects of pH

he effect of pH on the % removal of Pigment (PPD)from the Cadmium oxide nanoparticle surface was investigated at pH (3, 4, 5, 6, 7, 8, and 9) at 298 K, 90min contact time, and an adsorbent dose (0.05 g). The effects of pH on tincture (PPD) are shown in Figure 11. It can be seen that at a pH of 5, the elimination of Pigment (PPD) Where it reached the highest rate of adsorption by about (97%), and then began to decline as the value of the acidity function increased.

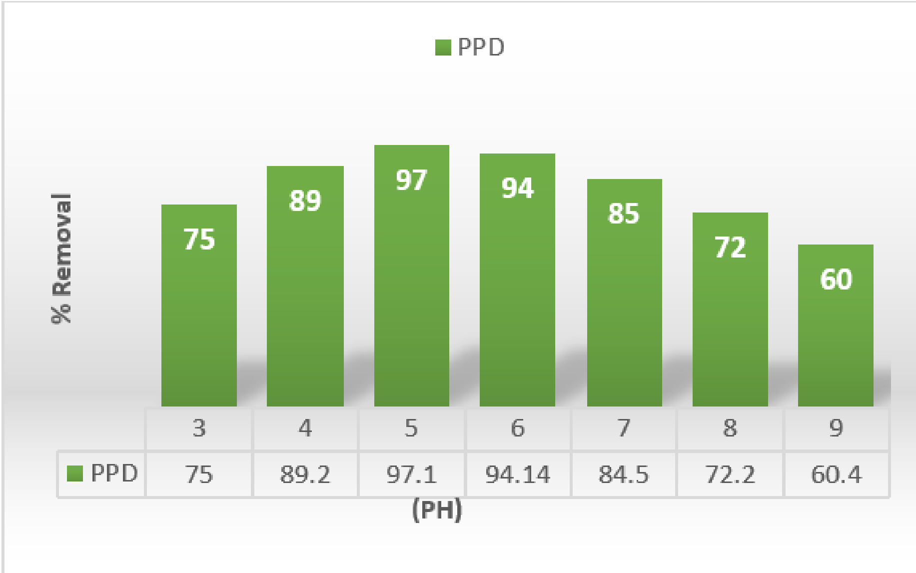

Figure (11): The effect of (PH) on the adsorption of Pigment (PPD) on the (CdO-NPs)

3.6.5 Temperature effect

At different temperatures (298, 308, 318, 328, and 338 K) and pH 6.5, we explain the influence of temperature on the amount of dye adsorption (PPD) The initial concentration was (100ppm) and the contact time was 90 minutes, and the amount used was (0.05g (Figure (12) shows the results obtained, as it was found that the increase in temperature increases the percentage of removal in relation to the (PPD) pigment, but the increase is slight and the reason is due to the fact that the increase in temperature will lead to the liberation of the functional groups and thus the viscosity of the solution decreases, which leads to an increase in the pores on the surface. The surface of the adsorbent material and the kinetic energy of the adsorbent molecules will also increase, and thus the rate of diffusion of the adsorbent molecules will increase, and the adsorption will increase [24].

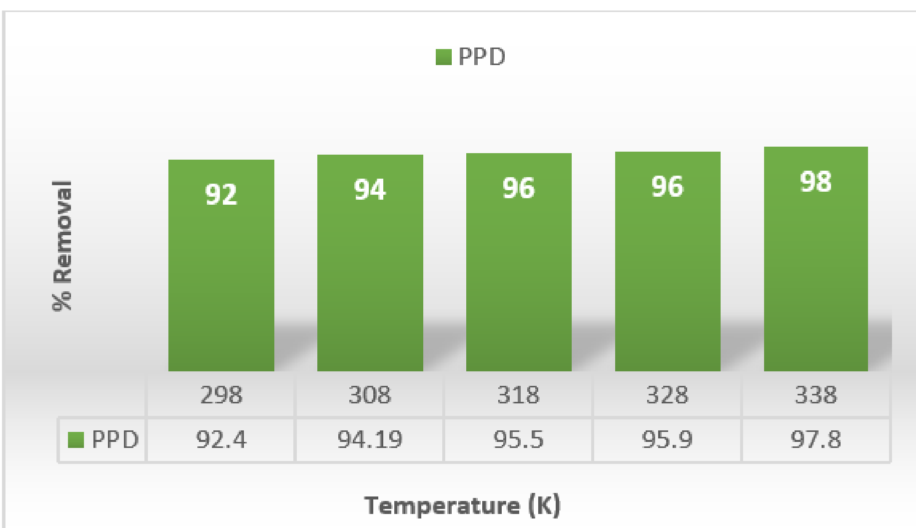

Figure (12): Effect of temperature on adsorption of Pigment (PPD)on (CuO- NPs)

3.6.6 Langmuir Equation

The Langmuir equation for adsorption of tincture (PPD).a dye was applied to the previously prepared adsorbent surface, which is Cadmium oxide nanoparticles (CdO -NPs) prepared by the green method at a temperature of (298 K) and from the slope and intercept we plot (Ce/Qe vs. Ce) As shown in the figure (13) then the Langmuir constants were extracted as shown in the table (2) according to the following equation.

$$\frac{Ce}{Qe} = \frac{1}{Qmax.k1} + \frac{Ce}{Qmax}$$

The slope and cut-off of the linear connection between Ce against Ce/Qe were used to get the values of Qmax and Kl.

3.6.7 Friendlich isotherm

The linear Friendlich equation was utilized to apply the adsorbents' practical adsorption findings to the utilized adsorbent surfaces.

$$LnQe = LnKf + \frac{1}{n}LnCe$$

The slope and cutoff linear connection between LnQe and LnCe were used to calculate the Freundlich constants n and K_f, as well as the value of the correlation coefficient R^2 for the adsorption process. And Figure (14) is the graph obtained when Lin Qe is plotted against Lin Ce to extract the Friendlich constants.

Table 2: Freundlich and Langmuir constants on the surface of (CdO-Nps)

Adsorbent	Langmuir			Freundilch		
	R^2	$Q_{max \times 10^3}$	K_l	R^2	n	K_f
PPD	0.9964	1250.0	0.0108	0.9989	1.136	15.148

The results in Table (2) show that the Langmuir adsorption equation, which produced high linear correlation coefficients between the diverse adsorption processes on the constructed adsorbent surfaces, applies to them in part (0.9964). Also, the Friendlich equation applies to it, where it gives linear correlation coefficients between (0.9989). From the results, we find that the values of (n>1), which represents the intensity of adsorption, which indicates that the adsorption that occurs is physical adsorption [24]. This is what happened with us upon the adsorption of the two ions shown in our study

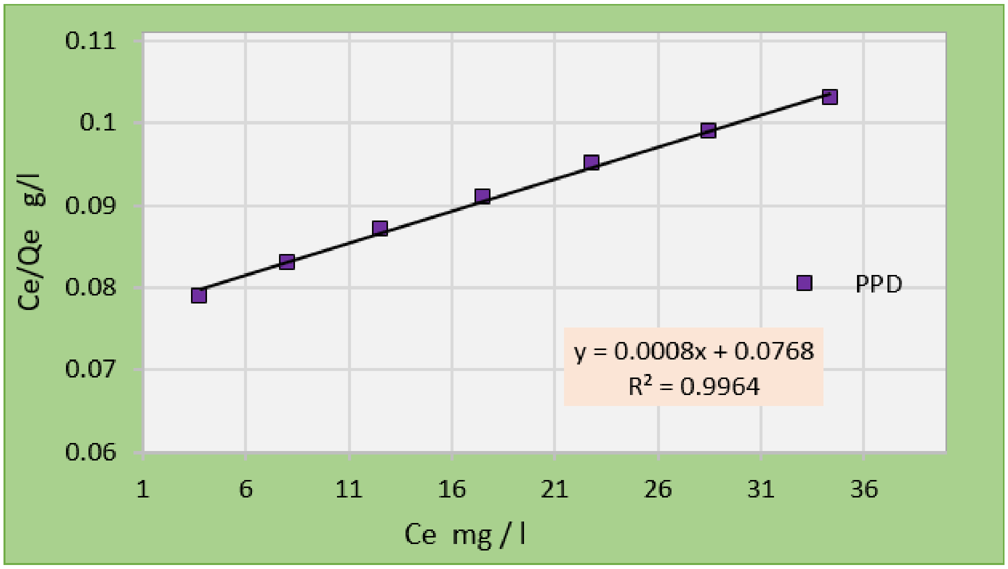

Figure (13): Langmuir isotherm to remove (PPD) on the surface of (CdO-NPs)

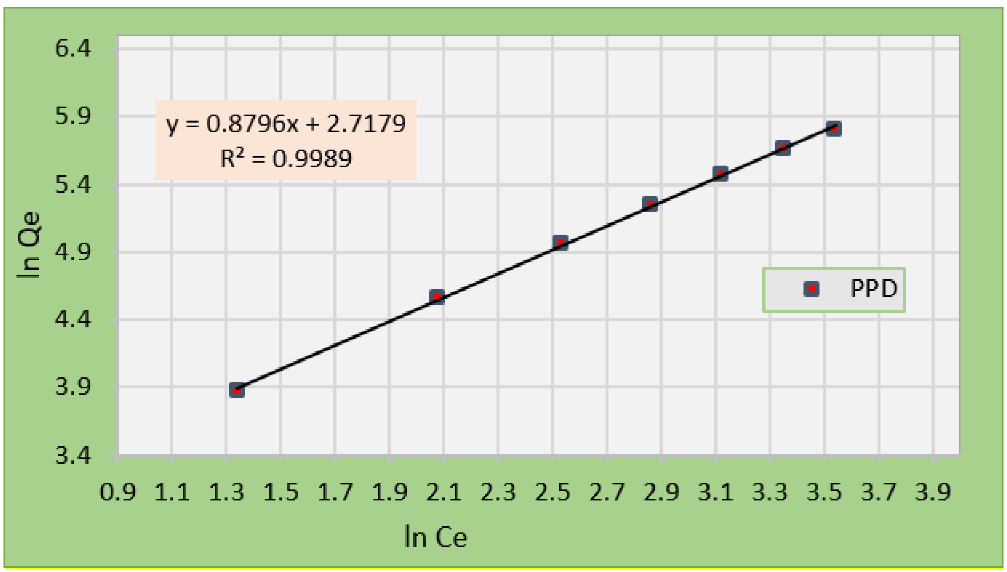

Figure (14): Freundlich isotherm to remove (PPD) on the surface of (CdO-NPs)

3.6.8 Thermodynamics

The thermodynamic functions of adsorption were calculated for tincture (PPD). Using nanoparticles of Cadmium oxide prepared by the green method. The content values for adsorption and adsorption entropy (ΔS) were calculated using the Vant Hoff equation [25].

$$LnK = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad , \quad K = \frac{Qe}{Ce}$$

The slope and cut-offs of the straight line equation were calculated from the graph of Ln k vs. 1/T, and the values of ΔH and ΔS were calculated from the slope and cut-offs of the straight line equation, respectively. Gibbs' mathematical equation was also used to compute the value of the free energy G.

$$\Delta G = \Delta H - T \cdot \Delta S$$

Table (6) show that the heat of adsorption (ΔH) is positive, indicating that all adsorption processes are endothermic, and that all values were less than (40 kg/mol), that adsorbent surfaces is physical. 23 Negative (G) values suggest that adsorption processes happen on spontaneously. The positive values of (ΔS) imply that the adsorbed molecules are less regular on the adsorbing surface than they are in the solution.

Table 3: Thermodynamic functions of adsorption on the surface of (CdO-NPs)

T_K	PPD		
	ΔG	**ΔH**	**ΔS**
298	**-6.96**	**-33.4**	**-86.7**
308	**-6.04**		
318	**-5.89**		
328	**-4.14**		
338	**-3.42**		

The obtained results indicate that the enthalpy values of adsorption (ΔH) were negative with both tinctures (PPD). and therefore the adsorption processes are exothermic and that all values were less than (40 kj / mol), which is an indication of The adsorption processes on the characteristic surfaces are of a physical nature [25]. The values of free energy ΔG indicate that all processes occur spontaneously because all values were negative. While the resulting values of (ΔS) for the two adsorbed ions, their values were negative, and this is evidence that the processes are less random, which proves that the adsorbed molecules are more regular on the adsorbent surfaces.

4. Conclusion

Tomato leaves extract and Cd $(NO_3)_2$ metal salts are used to synthesize Cadmium nanoparticles, which is a simple and environmentally friendly process. This method has numerous benefits, including economic viability, ease of scaling up, and reduced time consumption. Cadmium nanoparticles prepared in this way can be used as active surfaces to separate and remove tincture (PPD) from polluted water.

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