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By Universitas Muhammadiyah Sidoarjo

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Magnetic Nanocomposites Revolutionize Heavy Metal Adsorption for Environmental Cleanup

Nanokomposit Magnetik Merevolusi Adsorpsi Logam Berat untuk Pembersihan Lingkungan

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Abstract

General background: Magnetic nanocomposites have garnered significant attention due to their multifunctional properties, particularly in environmental remediation, where they can be used for the removal of heavy metals from aqueous solutions. **Specific background:** Polypyrrole (PPy) and poly(p-hydroxyaniline) (P(p-OH An)) are conductive polymers known for their adsorption capabilities, while Fe₃O₄ nanoparticles exhibit magnetic properties, facilitating separation and recovery. **Knowledge gap:** Despite the potential of Fe₃O₄-based composites, few studies have systematically explored the synergistic adsorption properties of PPy and P(p-OH An) in Fe₃O₄-based nanocomposites under varying environmental conditions. **Aims:** This study aimed to synthesize and characterize Fe₃O₄/PPy/P(p-OH An) magnetic nanocomposites and evaluate their adsorption performance under different temperatures and isotherm models. **Results:** The nanocomposite was synthesized through chemical oxidation polymerization and characterized using FTIR, TEM, AFM, and TGA, confirming its successful formation and nanoscale structure. Adsorption studies indicated an exothermic process, with a decrease in adsorption capacity at higher temperatures. The adsorption data fit the Freundlich isotherm better than the Langmuir model, suggesting heterogeneous surface adsorption. **Novelty:** This study demonstrates a novel Fe₃O₄/PPy/P(p-OH An) nanocomposite with superior adsorption properties, showing its potential in heavy metal ion removal and offering an improved understanding of temperature effects on adsorption performance. **Implications:** The findings underscore the composite's promise for environmental remediation applications, particularly in water treatment, and suggest further optimization of the adsorption conditions and evaluation of the composite's reusability for industrial-scale applications.

Highlights:

Enhanced Adsorption: Fe₃O₄/PPy nanocomposites offer combined magnetic and polymer adsorption properties.

Temperature Sensitivity: Adsorption decreases as temperature rises, indicating exothermic behavior.

Surface Interaction: Freundlich isotherm shows adsorption occurs on heterogeneous surfaces.

Academia Open

Vol 9 No 2 (2024): December

DOI: 10.21070/acopen.9.2024.10207 . Article type: (Engineering)

Keywords: Magnetic nanocomposite, Fe₃O₄, Polypyrrole, Adsorption, Environmental remediation.

Published date: 2024-09-24 00:00:00

Introduction

Nanocomposite polymers The word "polymer nanocomposite" has evolved to describe a system consisting of several components, the main one being a "polymer" or a mixture of them, and the minor component having the smallest "one dimension under" (100 nm) [1]. Materials with nano-scopic inorganic or organic particles dispersed in a "organic polymer matrix" are what significantly improve the polymer's physical characteristics. Because "nanocomposites" have length scales of nanometers, which lessen light dispersion, they are often transparent. "Nano-composites" are a new alternative to traditionally filled polymers

. Filler dispersion nanocomposites have significantly better characteristics than pure polymers or their traditional composites due to their nanoscale diameters [2].

These include enhanced modulus and strength, exceptional barrier qualities, enhanced resistance to heat and solvents, and decreased flammability [3]. The term "polymer nanocomposites" refers to a novel range of reinforcing materials that combine the manufacturing and processing pliability inherent in plastics, thermosets, and resins with new functions and uses for "polymers" [4]. High industrial importance materials are "polymer nanocomposites." Without scattering, nanosized particles enable improved mechanical and/or electrical properties. most notably, the polymer's tiny size preserves its optical clarity(5)

Methods

Raw Materials

Para- hydroxy aniline, pyrrole and Ethanol (sigma Aldrich). Iron (II) Chloride (ALPHA), Hydrochloric acid (BDH), Sodium dodecyl sulphate (CDH),Aluminum hydroxide (Merck), Nitrogen Gas (Industrial), Potassium Persulfate (CDH).

Preparation of Fe₃O₄ Nanoparticles

The following is how the "chemical precipitation method" was used to create Fe₃O₄ nanoparticles: A 100 ml round-bottom flask with four necks, a nitrogen gas input, a condenser, and a mechanical stirrer was filled with 40 ml of "deionized water." Subsequently, a 2:1 molar ratio of FeCl₃·6H₂O and FeCl₂·4H₂O were introduced into the flask. After 30 minutes of stirring the mixture in a nitrogen environment, 15 milliliters of NH₄OH aqueous solutions were added until the reaction mixture's pH reached 8. After that, the reaction was let to proceed at this pH for two hours.

. Finally, the resulting magnetic nanoparticles were dialyzed and purified repeatedly by magnetic field separation, and disposed by FeCl₃ solution (6)

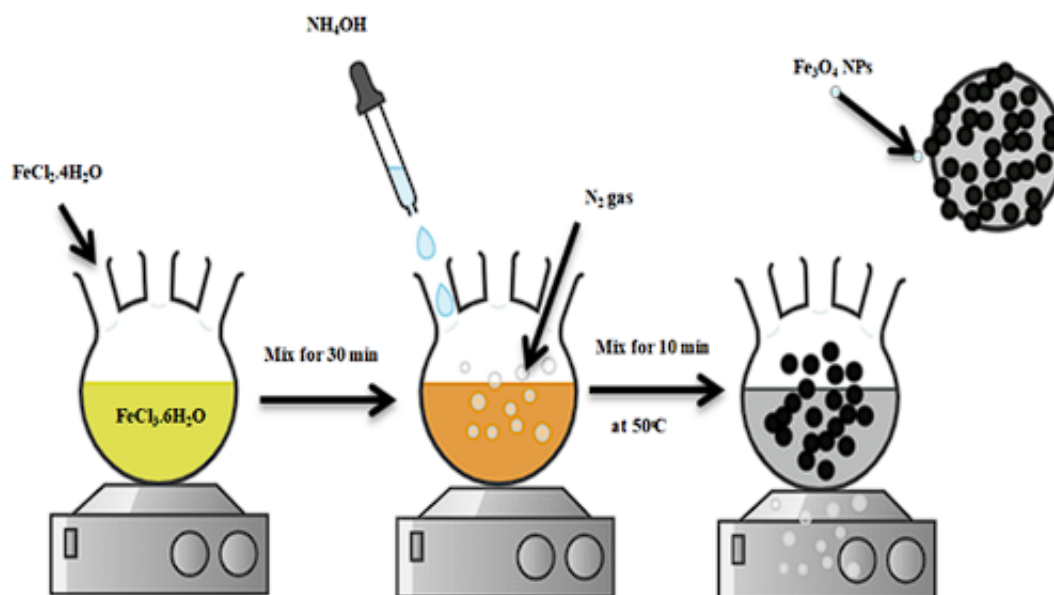


Figure 1. Synthesis Fe₃O₄ Nanoparticles

Synthesis of PPy-Fe₃O₄ Composites

The Fe₃O₄ nanoparticles that were made in the preceding stage were put into a 100 ml flask with a round bottom and a mechanical stirrer. At room temperature, 100 ml of deionized water was mixed vigorously to dissolve pyrrole monomer and dodecylbenzene sulfonic acid (NaDBS) in the flask. The mixed solution was supplemented with an acidic aqueous FeCl₃·6H₂O solution. After that, the polymerization was let to continue for 12 hours at room temperature while being stirred. The products underwent multiple washings in deionized water and methanol before being vacuum-dried for 24 hours at 60 °C (7).

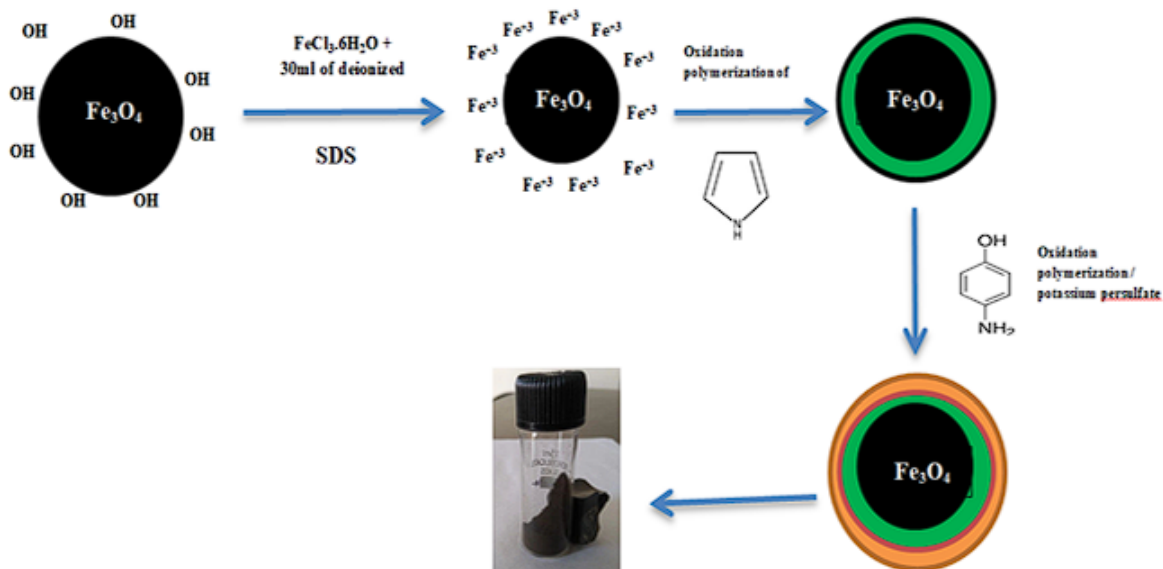


Figure 2. Synthesis of [Fe₃O₄/ poly pyrrole /poly (p-hydroxyaniline) Nanocomposite

PPy Synthesis Using FeCl₃ and Sodium Dodecyl Sulphate

SDS, or sodium dodecyl sulphate, was the surfactant employed in this method. De-ionized water (three milliliters) was mixed with one gram of SDS. To get a clear solution, the mixture was agitated for thirty minutes. One milliliter of pyrrole monomer was then added to the SDS solution drop by drop. FeCl₃ solution with the necessary molarity was added dropwise after the monomer was introduced, and it was left to polymerize for three hours at room temperature. The resulting black polymer powder was filtered and repeatedly cleaned with a solution of ethanol and de-ionized water. For a full day, the powdered polymer was dried in a vacuum oven. Another reaction was conducted using the same protocol as previously reported, with the exception that the polymerization period was extended to five hours (8) in order to examine the impact of time on the quality and yield of PPy produced

Synthesis of P (p-OH An)

The synthesis of P(p-OH An) involved dissolving 1g of para hydroxy aniline in 50 ml of HCl [0.1M], which was then kept at 5°C. Another solution was made by dissolving 1g of KIO₃ in 50 ml of HCl [1M], which was then added drop by drop to the first solution using a magnetic stirrer and stirred for 2 hours. The mixture was then kept at 5 °C for the second day and stirred once more for four hours. After stopping the churning of the solution and letting it sit for an hour, it was filtered and repeatedly cleaned with deionized water, yielding a glossy dark result. Three milliliters of hydrochloric acid, enhanced with ionized water to 100 ml this is for the purpose of increase doping it finally, dry in oven 40-50° C[9].

Synthesis of Fe₃O₄/PPy and P(p-OH An)

Dissolve 1 gram of Fe₃O₄/pyrrole in 100 ml in 0.1M HCL in an ultrasonic device for 30min We dissolve para hydroxy aniline it in 50 ml of 0.1M HCL. We dissolve potassium persulfate in 50 ml of 0.1M HCL We add the second solution to the first solution and leave for 15 minutes. After that we add the third solution to the first solution. Leave it for two hours, until a "black precipitate

' is formed. Wash it several times with" water and ethanol" .

Preparation of Standard Solutions and Analytical Method:

Standard Solutions

For the purpose of conducting analytical studies, the weights shown in Table (1) were used, and then Reserve standard solutions were prepared at a concentration of (1000 ppm) and were called storage solutions. (Solution Stock) Ions of the elements used in the study, which are (Ni+2) (From there, other standard solutions were prepared with a concentration of 100 parts per million (100 ppm) This is done by diluting certain volumes of standard reserve solutions with a concentration of (1000 ppm), where deionized distilled water was used all preparation processes

Metal	Molecular Formula	Molecular Weight	The Weight
Aqueous Nickel Nitrate	Ni(NO ₃) ₂ .6H ₂ O	290.80	4.954g

Table 1. Shows The Weight Used for The Compound

Analytical Measurement:

In this study, the meal method was used, which involves treating a specific weight of resin Study with specific volumes of solutions of the elements that are studied in this research in acid functions Specific and different time periods depending on the type and nature of the study, after which the solutions are filtered It is separated from the resin, and then the concentration of the remaining ions in the filtrate is determined by using Flame atomic absorption device, and thus through this method it is possible to determine the workability of the resin The method used in this study depends on the ions used in the research This method is summarized as follows Weigh (0.1 gm) of the prepared resin and put it in 10 ml volumetric bottles and add It has 10 ml of ion at a ppm concentration of 100 and at acid functions (1, 3, 5,7), then Volumetric bottles are placed in a water bath device equipped with a vibrator and a temperature regulator at a rate 120 r/min and at a temperature of 25 C° With different and specific time periods, 10 min , 30 min , 60 min , 90 min , 120 min , 150 min. After completion, the solutions were filtered and the concentration was calculated. The remaining ion in the filtrate solution using atomic absorption technology Atomic absorption without flameless

Result and Discussion

3-1: FT-IR Spectra of Fe₃O₄ Nanoparticles:

In Figure1)), the peak at ~3448.84 cm⁻¹ is attributed to the stretching vibrations of OH, which is assigned to " OH absorbed" by Fe₃O₄ nanoparticles." And the peak at 582.82 cm⁻¹ is attributed to the Fe-O bond vibration" of Fe₃O₄(10) .

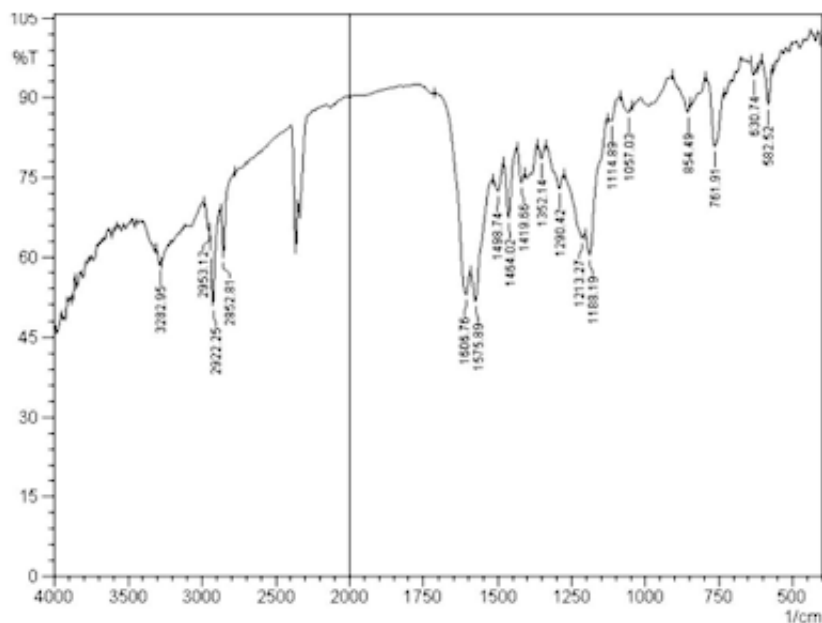


Figure 3. FT-IR of Fe₃O₄

3-2: FT-IR Spectra of Fe₃O₄/Ppy/P(p-OH An)

Figure (4) the wave numbers 3533.71cm⁻¹, the wave number shows the state of the O-H group(10). cm⁻¹. The absorption bands at wave numbers 1610.61cm⁻¹, 1573.97cm⁻¹, 1518.03cm⁻¹, represent the state of the Polypyrrole group that has been successfully combined with Fe₃O₄, the other peak at 1464.02 cm⁻¹ showed a decrease in the sharpness of the absorption peak, which indicated that PPy had been successfully composed with Fe₃O₄ nanoparticles. The absorption bands at wave numbers 933.58cm⁻¹, 895.00cm⁻¹, 839.06cm⁻¹, represent the state of the C=C group.

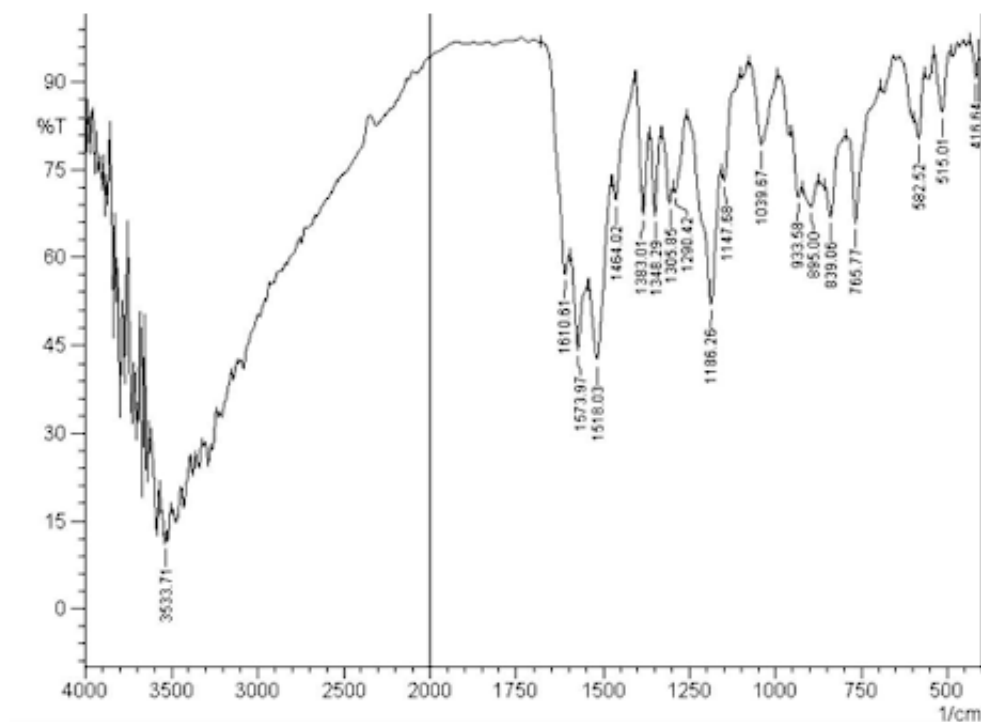


Figure 4. FT-IR of Fe₃O₄/Poly pyrrole /Poly (p-hydroxy aniline)

-poly(4-aminophenol) nanocomposite in presence of Fe₃O₄ as a ferromagnetic material exhibit new absorption peaks distinctly at 1573.97cm⁻¹, 1464.02cm⁻¹, 1348.01cm⁻¹, 1348.29cm⁻¹ and 1305.85cm⁻¹ which are “assignable to the presence of various metal oxides in the composite. the broad peaks at 3200-3500 cm⁻¹ are O-H Phenolic group in the nanocomposite”(12,13,14).

3-3: The TEM Photographs of Fe₃O₄ Nanoparticles

were shown in Figure (3) The Fe₃O₄ nanoparticles, the diameters of Fe₃O₄ particles were no more than 50 nm. showed the naked ones. The above result can be explained by the common ion effect that Fe⁺³ ion was easily absorbed onto the surface of Fe₃O₄ nanoparticles to form surrounding positively charged (Fe⁺³) shells, and the Fe₃O₄ nanoparticles dispersed more effectively by the repulsion of the common ions. the Fe₃O₄ MNPs show homogeneously spherical shape with diameter about 50nm(15).

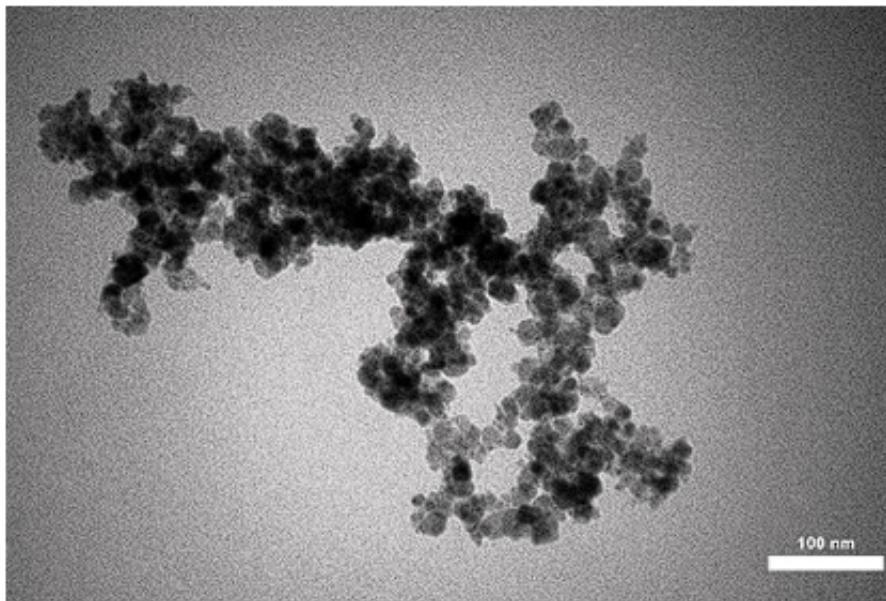


Figure 5. TEM of Fe₃O₄ Nanoparticles

3-4: The TEM Photographs of Fe₃O₄/Ppy/P(p-OH An)

Figure (4) of the poly(Ppy-P(p-OH An))/Fe₃O₄ nanocomposite showed a comparatively spherical morphology with Fe₃O₄ nanoparticles distributed throughout the copolymer matrix. The copolymer enveloped every Fe₃O₄ nanoparticle. Because of the difference electron penetrability, the lighter-contrast copolymer was deposited onto the darker-contrast Fe₃O₄ nanoparticles.(15)

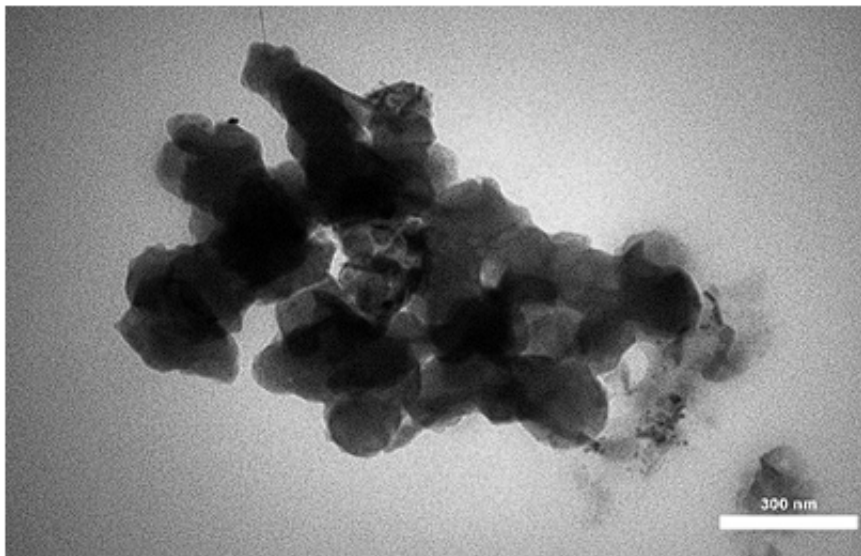


Figure 6. TEM of Fe₃O₄/poly pyrrole/poly(p-hydroxy aniline)

3-5: The AFM of Photographs of Fe₃O₄ Nanoparticles

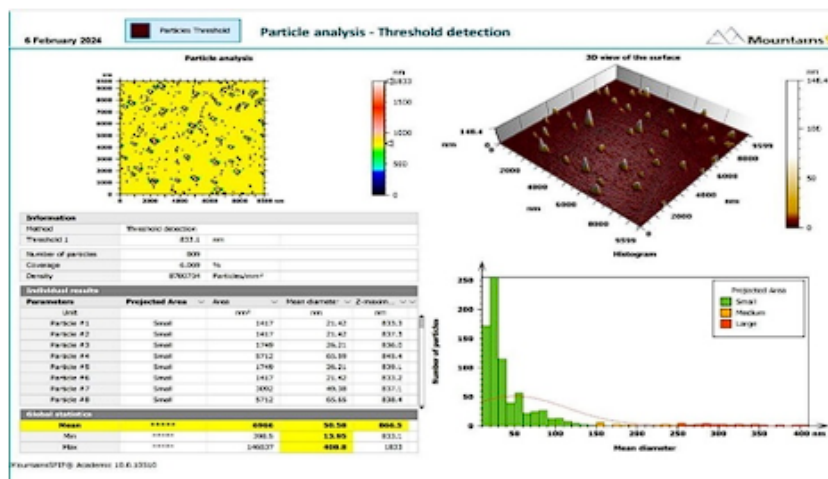
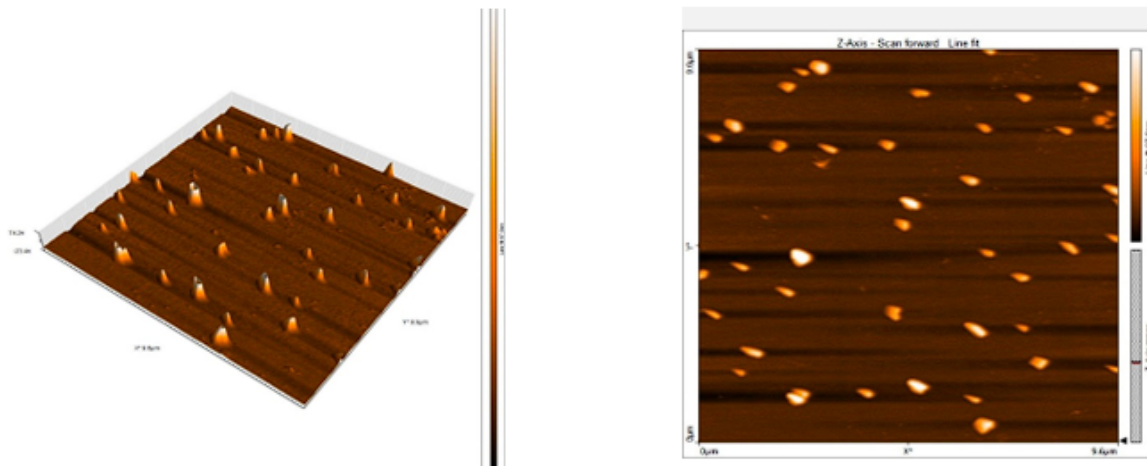


Figure 7. AFM Image Fe₃O₄ Nanoparticles

For AFM characterization, the magnetite nanoparticles were applied to the glass and allowed to dry. The information was gathered to ascertain the samples' roughness and three dimensions (3D). The generated three-dimensional images of the sample are displayed in Figure (7). Based on the histogram in Figure (7), the maximum height of the particles is around 3.492 nm, and their diameter is 50.58 nm for the scanned area of one by one.

The particle size determined by TEM and this result accord. According to [16], the yellow patches, or knobs, signify the presence of a tiny agglomeration of Fe₃O₄ nanoparticles. This is also visible as a yellow area at phase contrast in the 3D image. The sample was melted down as a result of heat absorption from the laser light; the bright yellow area is caused by the high moisture content in the ethylene glycol.

[17].

3-6: AFM of Fe₃O₄/Ppy/P(p-OH An)

The external surface of the co-polymer nanoparticles was displayed in Figure (8). The co-polymer surface's square root square was 8.981 nm, and its roughness coefficient was 6.125 nm. This suggests that the surface roughness, homogeneity, and homogeneous crystalline structure are all significantly influenced by the large size of the nanoparticles. Furthermore, the average particle height was found to be 552.4 nm, as shown in Figure 8. The results show that the molecular size of the co-polymer nanoparticle was 79.06 nm, and Figure (8) shows the distribution of the various proportions of co-polymer particle sizes. Table shows the total rate of the common nanoparticle particle sizes and the different proportions of these volumes.

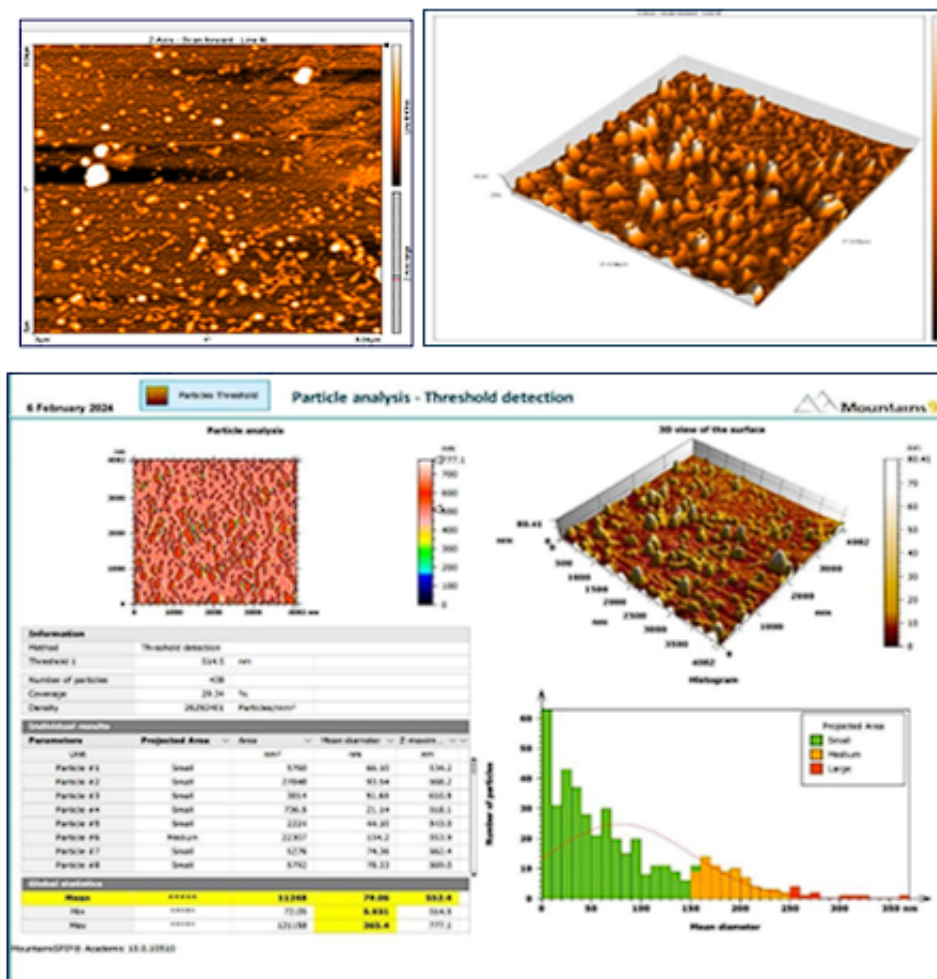


Figure 8. AFM Image of Fe₃O₄/Poly pyrrole/poly(p-hydroxy aniline)

3-7: Thermogravimetric analysis (TGA) of [Fe₃O₄/poly pyrrole / (p-hydroxyaniline)]:

TGA has demonstrated the copolymers' heat stability. The relative preview of the copolymers of (Fe₃O₄/polypyrrole/(p-hydroxyaniline)) in the TGA thermogram is shown in Figure (9) At 80.82 °C, the weight loss started, and it was around 3.975 percent. At around 152.90°C, there is a little increase in weight loss, approximately 5.023 percent, during the second weight loss event. Step three of the weight reduction process took place at 227.20 °C, or 11.31 percent. The base form of poly(p-hydroxy aniline) is quite stable up to about 152.90 °C, and it begins to break down at about 227.20 °C. The first weight loss in homo polymer pyrrole happened at around 80.82 °C, and the weight loss percentage was extremely low—about 3.975 percent. The second reduction in weight happened at (17). The disruption of the C-C structure is thought to be the cause of the polymers' abrupt breakdown. The rate of breakdown is nearly the same for both homopolymers and copolymers above 227.20 °C.

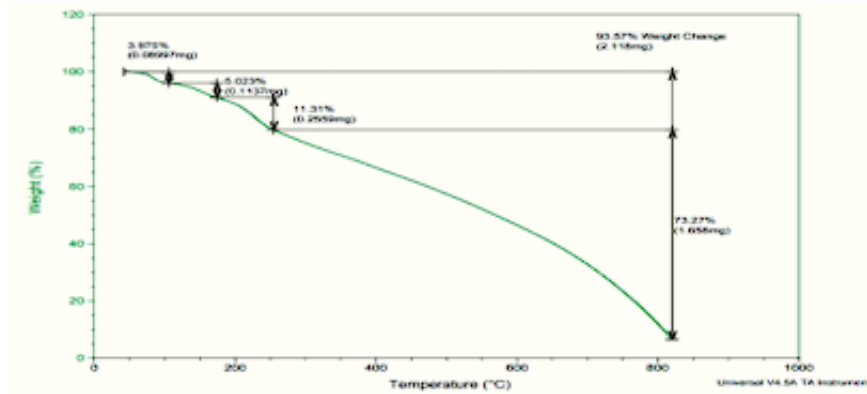


Figure 9. TGA of Fe3O4/Poly pyrrole /poly (p-hydroxy aniline) Nanocomposites

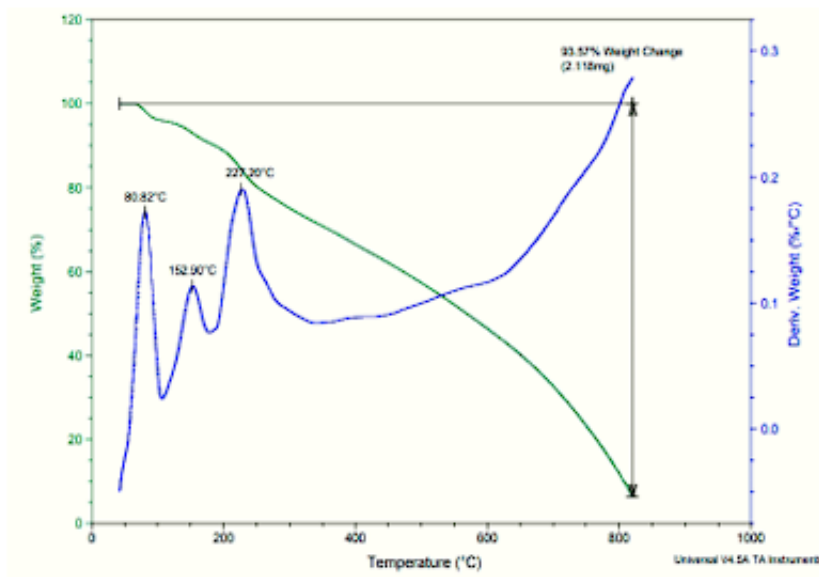


Figure 10. TGA of [Fe3O4/polypyrrole/(p-hydroxyaniline)] Nanocomposites

3-8: Adsorption Studies

3-8-1: Effect of temperature on adsorption

The adsorption of nano polymers prepared from their aqueous solutions was studied to remove some heavy elements at different temperatures (25, 40, 50) and at constant conditions of concentration (1000ppm) and volume (10ml). The results were recorded in tables (2), (3) and a graphical relationship was drawn between The amount of adsorbed material (qe) and time to obtain the general form of the adsorption isotherms.

3-8-2: Study of The Effect of pH on The Analytical Efficiency of Nano Polymers

prepared with the studied ions under conditions of constant concentration of 100ppm and temperature of 25 the)qe(is high in the acidic medium and this behavior is observed in this study .the reason for this is that in the acidic medium the number of hydrogen ions increases if there is a repulsion between the ions of the nano polymer that carry a positive charge and the hydrogen ions in the solution in association with the negative sites present on these adsorbent surfaces

pH= 7		pH=5		Ph=3		Ph=1		t(min)	Sorbent	Metal
%E	Qe	%E	Qe	%E	Qe	%E	qe			
91.98	9.19	75.49	7.54	64.74	6.74	14.43	1.44	10	Fe3O4/poly pyrrole /poly (p-hydroxy	Ni

92.28	9.22	79.67	7.96	64.74	6.47	11.12	1.11	30	aniline)	
91.78	9.17	71.89	7.18	66.84	6.68	11.91	1.19	60		
91.67	9.167	69.74	6.97	65.53	6.553	12.23	1.22	90		
91.62	9.162	67.78	6.77	66.58	6.658	11.59	1.15	120		
91.10	9.11	65.9	6.59	64.82	6.48	13.17	1.31	150		

Table 2. Adsorption Data at Different PH Values

%E	qe (mg/L)	P H
14.43	1.44	1
67.44	6.74	3
79.67	7.96	5
92.28	9.22	7

Table 3. Relationship Between %E and PH

%E	qe (mg/L)	T(K)	T(C)
92.28	9.22	298	25
59.89	5.98	313	40
54.89	5.48	323	50

Table 4. Relationship Between Temperature (K) and Capacity (qe)

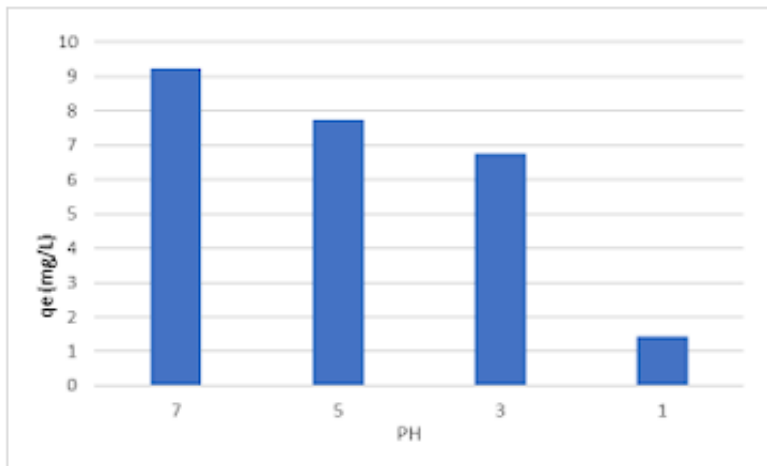


Figure 11. Relationship Between qe and PH

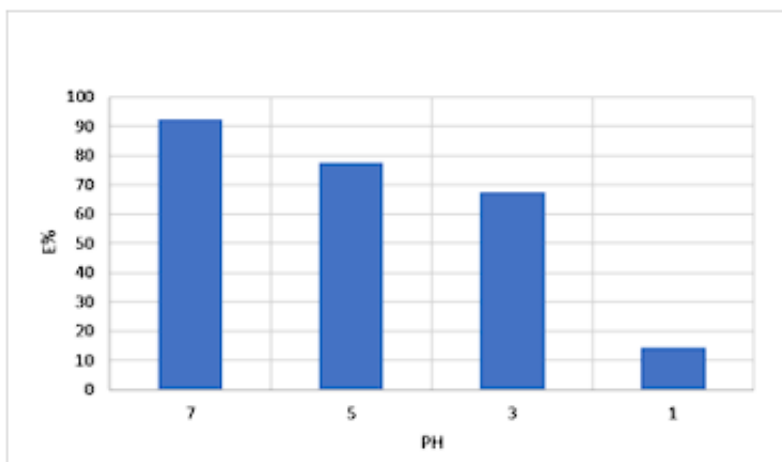


Figure 12. Relationship Between %E and PH

3-9: The Adsorption Isotherms :

Notice from these results in table (8) that the value of the correlation coefficient R2 for the Langmuir model at various temperatures is small values, as well as the appearance of negative values for the KL value, and this does not apply to the isotherm equation. As a result, we conclude that the Langmuir equation model does not apply to the Fe3O4/polypyrrole adsorption process. poly(p-hydroxy aniline) We note from the results in table (8) that the values of Freundlich constants (KF) increase with increasing temperature for the adsorbent polymer in this study and at different temperatures, and this is proof that the adsorption process is endothermic.

The value of the adsorption intensity (n) indicates the preference of the adsorption system and the degree of heterogeneity of the adsorbed surface. When its value is limited to (1-10), this means that the adsorption is good, and this is what we observed on the surface of (Fe3O4/poly pyrrole/poly (p-hydroxy aniline). (When the value of n is 10.570) and at 40 ° C 1.2) this is good adsorption, at 50 ° C there is poor adsorption. As for the values of the correlation coefficient R2 for the Freundlich model, we note the application of the equation to the surface of the adsorbed polymer. Thus, we conclude a partial application of the Freundlich equation to the nanopolymer adsorption process. This means that the adsorption process occurred on non homogeneous surfaces, meaning that it has different sites for adsorption energies.

3-10: The Langmuir isotherm model :

Adsorbent	298 K			313 K			323 K		
	KL	qemax	R2	KL	qemax	R2	KL	qemax	R2
Fe3O4/pyrrole/p-hydroxy aniline	-1.3819	8.3752	0.9997	-0.0491	2.9824	0.9973	-0.0297	1.9105	0.2348

Table 5. Langmuir Isotherm

3-11: The Freundlich model

Adsorbent	298 K			313 K			323 K		
	Kf	n	R2	Kf	n	R2	kf	N	R2
Fe3O4/pyrrole/p-hydroxy aniline	0.0621	-4.5913	0.0551	0.3197	-1.2233	0.9965	0.6853	-0.4270	0.0358

Table 6. Freundlich Isotherms

3-12: Calculation Thermodynamic Functions:

T(K)	ΔG°	ΔH°	ΔS°
298	-5.764	-239.563	-226.564
313	35.3910		
323	565.01278		

Table 7. Shows the values of thermodynamic equilibrium constants at different temperatures(Fe3O4/pyrrole/p-hydroxy aniline)

Conclusion

In this study, Fe3O4/polypyrrole/poly(p-hydroxyaniline) magnetic nanocomposites were synthesized using chemical oxidation polymerization and characterized by FTIR, TEM, AFM, and TGA. The FTIR spectra confirmed the successful incorporation of polypyrrole and p-hydroxyaniline with Fe3O4 nanoparticles, while TEM and AFM analyses revealed their homogeneously spherical morphology and nanoscale size distribution. The adsorption studies demonstrated that the process was exothermic, with a decrease in adsorption capacity at higher temperatures, and the Freundlich isotherm provided a better fit compared to the Langmuir model, indicating adsorption on heterogeneous surfaces. These findings suggest potential applications of the synthesized nanocomposites in environmental remediation, particularly for the removal of heavy metal ions from aqueous solutions. Future research should explore the optimization of adsorption conditions and the nanocomposites' stability and reusability for large-scale applications.

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