

## Kinetic, Isotherm Study of Nickle Adsorption from Colored Wastewater Using Polypyrrol/ Polyvinyl Alcohol Composite

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**Abstract:** Recent project deals to study the adsorption of Ni ions from colored wastewater using adsorbents composite polypyrrol based on polyvinyl alcohol. These experiments were conducted in a batch system. Identification of the synthesized adsorbents was performed by SEM (Scanning Electron Microscope) & FTIR (Fourier transform infrared). Effect of the experimental parameters pH, contact time and dose of adsorbent base on the surface adsorption of colored wastewater has been studied. According to results, optimum pH for absorption of Ni by mentioned absorbent was 3. Absorption increased with increasing contact time and after 14 min reached to equilibrium state. Optimum dose of adsorbents has been reported 0.4 grams. The obtained equilibrium data in the initial concentration of Ni in the studied temperature range has a good adaptation with Freundlich isotherm. Evaluation of experimental data Indicated that the surface adsorption of Ni follows the pseudo-second order equation.

**Key words:** Absorption % Freundlich isotherm % Pseudo-second order equation % Polyvinyl alcohol % Polypyrrol

### INTRODUCTION

Conducting polymer matrices provide an interesting and useful focus for expansion of the fields of polymeric reagent research and molecular engineering. They are electrically conductive which makes their use as metal replacement materials of some interest. Conductive electro-active polymers such as polypyrrole (PPy) possess some unique chemical and electrochemical properties. PPy is attractive as an electrically conducting polymer because of its relative ease of synthesis. Bulk quantities of PPy can be obtained as fine powders using the oxidative polymerization of the monomer by selected transition metal ions in water or various other solvents [1-3]. The insolubility in common solvents and infusibility of conducting polymers, in general, make them poorly processable either by solution technique or by melt processing methods [4, 5]. Improvement of these material properties can be achieved either by forming copolymers of pyrrole, or by forming PPy composites or blends with commercially available polymers or inorganic materials which offer better mechanical and optical

properties, stability and processability [4, 6]. A general route for creating PPy composites is the in situ polymerization of pyrrole in the presence of organic or inorganic substances. Other useful approach for the improvement of the processability and the mechanical properties of insoluble polymers is the blending with soluble matrix polymers. Blending is an important process for developing industrial applications of polymeric materials and compatibility among components has a marked influence on the resulting physical properties of polymer blends [7]. Blending improves the mechanical and thermal properties of the individual polymer. Poly(vinyl alcohol) is a non toxic, water soluble, synthetic polymer with its excellent film forming properties. While PVA itself has poor metal ion binding capacity, sorption properties of PVA can be enhanced by grafting with suitable binding sites. PVA is cheap and its physic- chemical properties can be enhanced by simultaneous cross- linking and grafting. The removal of toxic heavy metals such as cadmium, copper, lead, nickel, mercury and zinc from aqueous environment has received considerable attention in recent years due to their toxicity

and carcinogenicity which may cause damage to various systems of the human body. They also can be readily absorbed by marine animals and directly enter the human food chains, thus presenting a high health risk to consumers [8]. Nickel ions are non-biodegradable toxic heavy metals and may cause dermatitis and allergic sensitization [9-11]. According to the World Health Organization guidelines, the maximum permissible concentration of The removal of toxic heavy metals such as cadmium, copper, lead, nickel, mercury and zinc from aqueous environment has received considerable attention in recent years due to their toxicity and carcinogenicity which may cause damage to various systems of the human body. the maximum permissible concentration of nickel in effluents in the US from the electroplating process wastewater is 4.1 mg/l, while that in drinking water should be less than 0.1 mg/l [12].

The major sources of nickel contamination to water comes from industrial process such as electroplating, batteries manufacturing, mine, metal finishing and forging. Different methods were investigated and applied to remove nickel ions from water such as adsorption, chemical precipitation, ion exchange, filtration, membrane separation and reverse osmosis. Adsorption is widely used because it is cost-effective and simple [13-14].

In this study, removal of Ni(II) using adsorbents composite polypyrrol based on polyvinyl alcohol is studied. The effects of dosage of sorbent, contact time and pH value on sorption of Ni(II) are investigated in detail, also adsorption isotherms and adsorption kinetics are obtained.

## Experimental

**Instrumentation:** Magnetic mixer model MK20, digital scale model FR200, atomic absorption device perkin-elmer model 2380, pH meter model 211 HANNA, vacuum oven model Binder USA, differential scanning calorimeter model PL-DSC, fourier transform infrared (FTIR) spectrometer model shimadzu 4100 and scanning electron microscope (SEM) model LSM50A were employed.

**Reagents and Standard Solutions:** Materials used in this work were pyrrole ( $d = 0.97$  g/mL), ethyl acetate, poly(vinyl alcohol) (PVA,  $M_w = 72000$ ,  $d = 1.19-1.31$  g/mL), from Merck and Aldrich. All reagents were used as received without further purification, unless stated otherwise. Distilled deionized water was used throughout this work. Pyrrole monomer was purified by simple distillation.

## Synthesis of Polypyrrole/ Polyvinyl Alcohol Composite:

In a typical experiment 0.1 gr Poly vinyl alcohol was added to a stirred aqueous solution (100 mL) containing 5 g of FeCl<sub>3</sub> as oxidant when the solution became homogen with orange color after 10 min. After 15 min, 1 ml of polypyrrol was added to solution. After 5 hours, the composite was collected by filtration and in order to separate the oligomers and impurities, the product was washed several times in succession with deionized water. It was then dried in a vacuum oven at 40°C for 12 h [15].

## Method of Nickel

**Method of Nickel Removal:** Completely mixed batch reactor (CMBR) technique was used to remove nickel ions from water. For determining the amount of heavy metal removal by adsorbent, 100 ml of nickel solution was mixed with 1 g powder of adsorbent and then stirred using magnetic mixer for 30 min and then was filtered respectively. The nickel concentration was analyzed by atomic adsorption method.

In addition, for calibrating the atomic absorption device, four standard samples with concentration of 1, 2, 4 and 6 mg/L were prepared and the corresponding amounts of absorption for each concentration were measured. Then by considering the amount of absorption equivalent to each concentration the linear regression curve was drawn. After drawing the calibrating diagram, the samples were injected to the device and their absorption was calculated. Then by considering this diagram the nickel absorption determined for different adsorbents. All of the experiments were repeated two times.

## RESULTS AND DISCUSSION

### Characterization of Polypyrrole / Poly Vinyl Alcohol:

In Figs. 1 and 2, scanning electron micrographs of polypyrrole / poly vinyl alcohol composite were shown. The SEM micrographs characterized the morphology of the polymer. As can be seen, PVA as a stabilizing agent could affect the size, morphology and homogeneity of particles, because the surface active agents are adsorbed physically or chemically by the growing polymer.

Because of the surface active agents prevent gross aggregation of the particles, PPy particles synthesized by suspension polymerization (without PVA) are larger than the PPy particle, were obtained in the presence of PVA as a surface active agent.

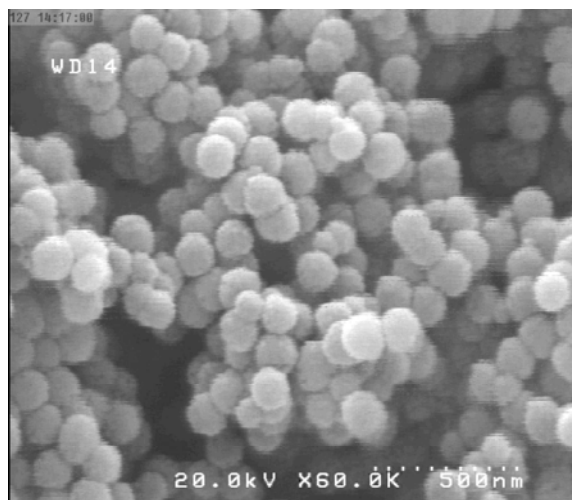


Fig. 1: SEM of Polypyrrole/PVA Nanocomposites Generated in Aqueous Media.

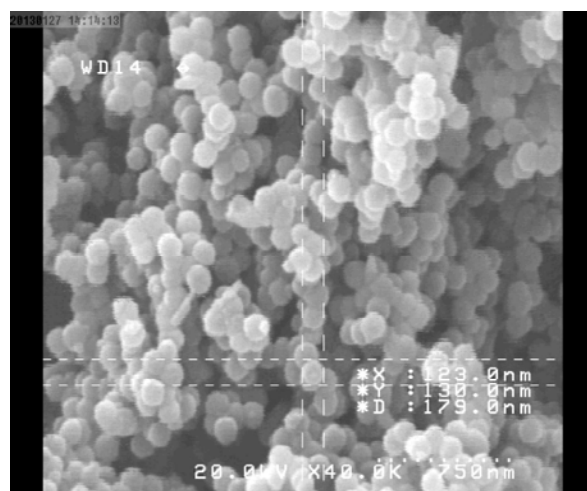


Fig. 2: SEM of Polypyrrole/PVA Nanocomposite Generated in Aqueous Media.

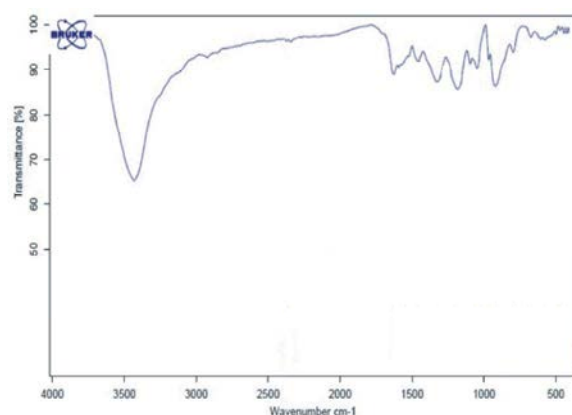


Fig. 3: FTIR Spectra of PPy in Aqueous Media.

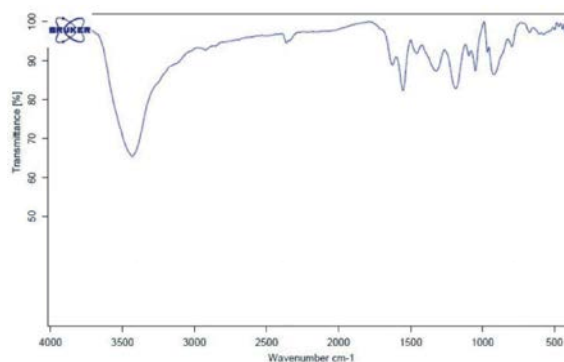


Fig. 4: FTIR Spectra of PPy / PVA (PVA used as stabilizing agent in Aqueous Media).

The chemical structure of the obtained products was determined by FTIR spectroscopy, which has provided valuable information regarding the formation of polypyrrole /poly vinyl alcohol composite. The FTIR spectra analysis has been done to identify the characteristic peaks of products. The FTIR spectra in the 2500-450  $\text{cm}^{-1}$  region for Ppy composites were shown in Fig. 3.

As can be seen, the FTIR spectrum changed when the composites were obtained in various conditions. As can be seen in Fig. 3 the peak related to pyrrole unit at 1547  $\text{cm}^{-1}$ . The peaks are at 1450  $\text{cm}^{-1}$  (C-N stretching vibration), 1292  $\text{cm}^{-1}$  (C-H in-plane deformation), 1037  $\text{cm}^{-1}$  (N-H in-plane deformation), 781  $\text{cm}^{-1}$  (C-H out-of-plane deformation) and 632  $\text{cm}^{-1}$  (C-H out-of-plane ring deformation). It can be seen in Fig. 4, the PVA presence intensity of peaks increased.

**Batch Adsorption Experiments:** The adsorption experiments in this work were done for the study the effect of experimental conditions on Ni(II) adsorption and determining the conditions that achieve the maximum amount of Ni(II) removal. Isotherm and kinetic evaluations were also conducted in this portion of the study. The adsorption tests were conducted in magnetic mixer. The magnetic mixer was 400 rpm throughout the study with 100 mL of solution prepared from the dilution of 1 g/L stock solutions. At the end of predetermined time intervals, the sorbate was filtered and the concentration of Ni(II) was determined. All experiments were carried out twice and the adsorbed concentrations given were the means of duplicate experimental results. The experimental error was below 4%, the average data were reported. The efficiency of Ni(II), % Removal, was calculated as:

$$\% \text{ Removal} = (C_i - C_f) / C_i \times 100$$

where  $C_i$  is the initial concentration ( $\text{mg L}^{-1}$ ) and  $C_f$  is the final concentration ( $\text{mg L}^{-1}$ ).

$q$  is the amount of metal adsorbed per specific amount of adsorbent ( $\text{mg/g}$ ). The sorption capacity at time  $t$ ,  $q_t$  ( $\text{mg/g}$ ) was obtained as follows:

$$q_t = (C_i - C_t) \times V / m$$

where  $C_i$  and  $C_t$  ( $\text{mg/l}$ ) were the liquid-phase concentrations of solutes at initial and a given time  $t$ ,  $v$  was the solution volume and  $m$  the mass ash ( $\text{g}$ ). The amount of adsorption at equilibrium,  $q_e$  was given by:

$$q_e = (C_i - C_e) \times V / m$$

where  $C_e$  ( $\text{mg/l}$ ) was the ion concentration at equilibrium.

**Effect of pH Sorption:** The pH of the solution affects the charge on the surface of the adsorbents, so the change in pH also affects the adsorption process and the  $\text{H}^+$  ion concentration may react with the functional groups on the active sites on the adsorption surface. In general, adsorption of cations is favored at  $\text{pH} > \text{pH}_{\text{pzc}}$ . The pH of the solutions has been identified as the most important variable governing metal adsorption. This is partly due to the fact that hydrogen ions themselves are strong competing ions and partly that the solution pH influences the chemical speciation of the metal ions as well as the ionization of the functional groups onto the adsorbent surfaces. In order to evaluate the influence of this parameter on the adsorption, initial Ni(II) concentration was of 40 ppm. Also PPY/ PPA composite dose of 0.5 g in 100 ml were used. the experiments were carried out at different initial pH values from 2 to 10. The effect of pH on adsorption efficiencies are shown in Fig. 5. Optimum  $\text{pH}=3$  is observed and the sorption rate reaches up to 91.13 % when contact time is 20 min for the Ni(II). when pH increases, there is a decrease in positive surface charge (since the deprotonation of the sorbent functional groups could be occurs), which results in a lower electrostatic repulsion between the positively charged metal ion and the surface of PPY/ PPA composite, favoring adsorption.

**Effect of Contact Time:** Figure 6 shows the effect of contact time on sorption of Ni(II) by of PPY/ PPA composite. For these cases, initial Ni(II) concentration

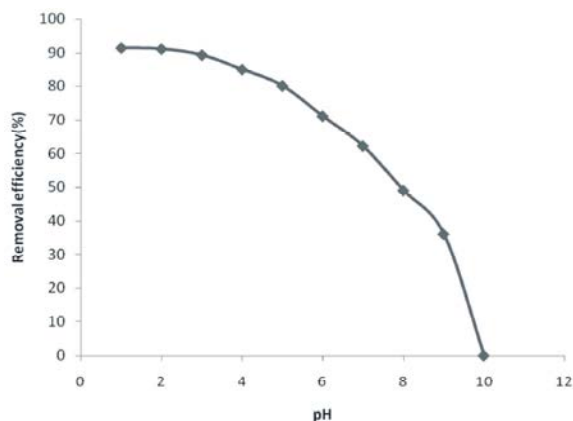


Fig. 5: Effect of pH on adsorption efficiencies

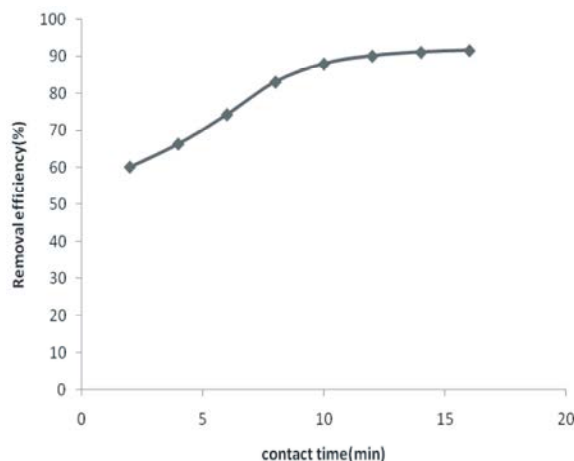


Fig. 6: Effect of contact time on adsorption efficiencies

were of 100 ppm, pH of 3 was used for Ni(II). Also PPY/ PPA composite dose of 0.5 g in 100 ml were used. For Ni(II), sorption rate reaches up to 91.2 % when contact time is 14 min and then little change of sorption rate is observed. This result revealed that adsorption of Ni(II) is fast and the equilibrium was achieved by 14 min of contact time. Taking into account these results, a contact time of 14 min was chosen for further experiments.

**Effect of PPY/ PPA Composite Dosage on Sorption of Ni(ii):** The effect of PPY/ PPA composite dose was studied for a by varying the dose between 0.05 g and 0.5 g in 100 ml aqueous. These tests were conducted at a temperature of 20 °C, with optimum pH value for Ni(II). The initial metal ion concentration was 40 ppm. It was observed that the adsorption percentage of Ni(II) onto the PPY/ PPA composite increased rapidly with the increasing of adsorbent concentration (Fig. 7). This result is expected because the increase of adsorbent dose leads to greater surface area. When the adsorbent

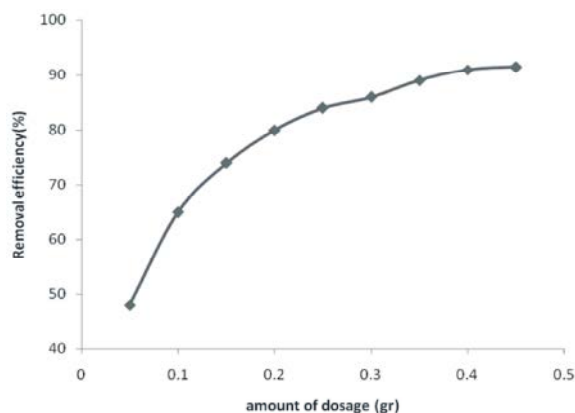


Fig. 7: Effect of amount of dosage on adsorption efficiencies

concentration was increased from 0.05 g to 0.4g, the percentage of Ni(II) ions adsorption increased 91.1. At higher concentrations, the equilibrium uptake of Ni(II) did not increase significantly with increasing PPY/ PPA composite. Such behavior is expected due to the saturation level attained during an adsorption process. For subsequent studies, a dose of 0.4g of PPY/ PPA composite into 100 ml aqueous solution was selected.

**Kinetics of Sorption:** Most of the adsorption transformation processes of various solid phases are time dependent. To understand the dynamic interactions of Ni(II) with ash and to predict their fate with time, knowledge of the kinetics of these processes is important [16].

Various kinetic models, namely Morris-Weber, Lagergren rate expression and pseudo second order models have been used for their validity with the experimental adsorption data for the Ni onto PPY/ PPA composite. It was assumed to offer no mass transfer (both external and internal external) resistance to the overall adsorption process. Therefore kinetic can be studied through the residual metal ion concentration in the solution. The study of adsorption kinetics describes the solute uptake rate and evidently these rate controls the residence time of adsorbate uptake at the solid-solution interface including the diffusion process. To investigate the change in the concentration of sorbate onto sorbent with shaking time, the kinetic data of Ni(II) ions sorption onto PPY/ PPA composite were subjected to Morris-Weber (intraparticle)Eq. (1) [17]:

$$q_t = K_{id}(t)^{0.5} + C \quad (1)$$

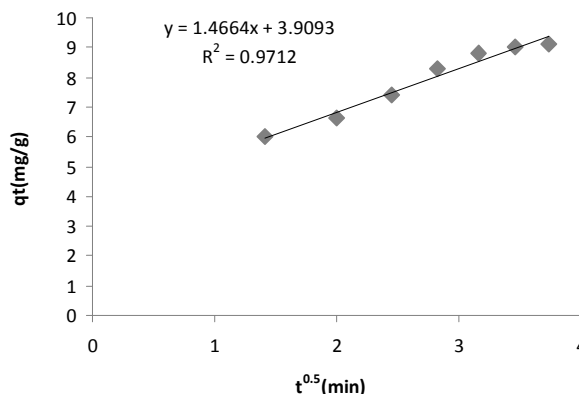


Fig. 8: Morris-Weber plot of Ni(II) sorption onto PPY/ PPA composite (the initial concentration, pH, volume of solution and amount of adsorbent was 40ppm and 0.4 g respectively).

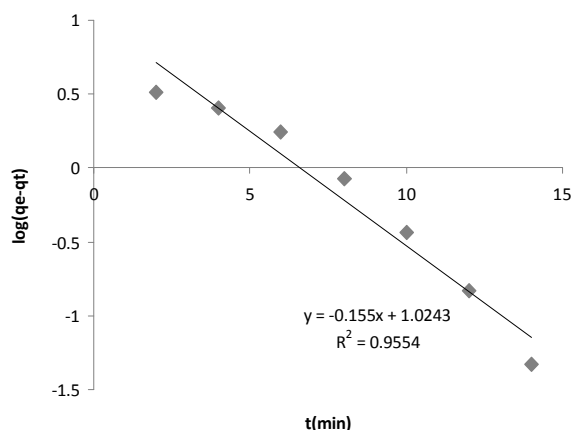


Fig. 9: Validation of Pseudo first order plot of Ni(II) sorption onto PPY/PPA composite (the initial concentration, pH, volume of solution and amount of adsorbent was 40 ppm, 3, 100 ml and 0.4 g respectively).

where  $q_t$  is the sorbed concentration of Ni(II) ions at time ' $t$ '. The plot of  $q_t$  versus  $t^{1/2}$  is given in Fig. 8. The value of rate constant of Morris-weber transport,  $K_{id}$ , calculated from the slope of the linear plot are shown in Fig. 8. The rate constant  $k_{id} = 1.466 \text{ min}^{-1}$  was calculated from the slope of the straight line with a correlation factor of 0.971.

The Pseudo first order of the sorption of Ni(II) ions onto PPY/ PPA composite was evaluated by treating the data to the following form of Lagergren rate expression (2) [18], to determine the rate constant of sorption for Ni(II) ions-PPY/ PPA composite system.

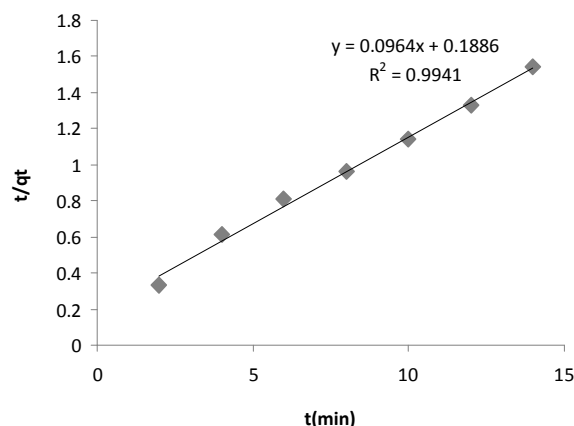


Fig. 10: Validation of Pseudo second order plot of Ni(II) sorption onto PPY/ PPA composite (the initial concentration, pH, volume of solution and amount of adsorbent was 40 ppm, 3, 100 ml and 0.4 g respectively).

$$\log(q_e - q_t) = \log q_e - \left( \frac{k}{2.303} \right) t \quad (2)$$

where  $q_e$  is the sorbed concentration at equilibrium and  $k$  is the first order rate constant. The linear plot of  $\log(q_e - q_t)$  against time 't' (Fig. 9) demonstrates the applicability of the above equation for Ni (II) ions sorption onto PPY/ PPA composite. The rate constant  $k = 0.3569 \text{ min}^{-1}$  was calculated from the slope of the straight line with a correlation factor of 0.995.

The kinetic data of Ni(II) ions sorption onto PPY/ PPA composite was subjected to pseudo second order Eq. (3) [19]:

$$\frac{t}{q_t} = \frac{1}{Kq_e^2} + \frac{t}{q_e} \quad (3)$$

The rate constant was calculated from the slope of the straight line (Fig.10). The rate constant was calculated from the slope of the straight line (Fig.10). The rate constant  $k = 0.049 \text{ min}^{-1}$  was calculated from the slope of the straight line with a correlation factor of 0.994. Also this suggests the assumption behind the pseudo-second-order model that the Ni(II) uptake process is due to chemisorptions [20]. The assumption behind the pseudo-second-order kinetic model was that the rate-limiting step might be chemisorptions involving valence forces through sharing or exchange of electrons between adsorbent and adsorbate [21].

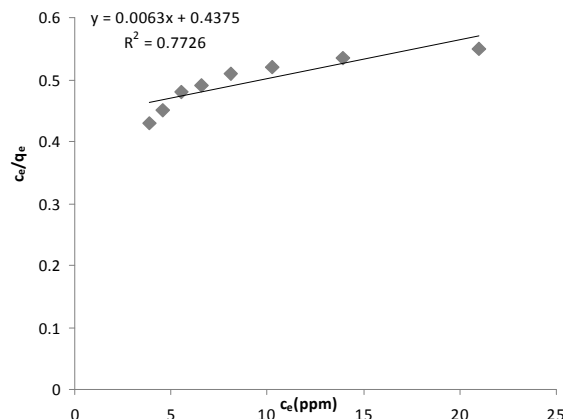


Fig. 11: Langmuir sorption isotherm for Ni(II) (the initial concentration, pH, volume of solution and contact time was 40 ppm, 3, 100 ml and 14 min respectively).

**The Isotherm Model:** The adsorption isotherm is based on the assumption that every adsorption site is equivalent and independent of whether or not adjacent sites are occupied. Isotherms show the relationship between metal concentration in solution and the amount of metal sorbed on a specific sorbent at a constant temperature.

**The Langmuir Isotherm Model:** The Langmuir isotherm model is valid for monolayer adsorption onto surface containing finite number of identical sorption sites which is presented by the following equation (4):

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (4)$$

where  $q_e$  is the amount of metal adsorbed per specific amount of adsorbent (mg/g),  $C_e$  is equilibrium concentration of the solution (mg/L) and  $q_m$  is the maximum amount of metal ions required to form a monolayer (mg/g). The Langmuir equation can be rearranged to linear form for the convenience of plotting and determining the Langmuir constants ( $K_L$ ) as below. The values of  $q_m$  and  $K_L$  can be determined from the linear plot of  $C_e/q_{eq}$  versus  $C_{eq}$ :

$$\frac{C_e}{q_e} = \frac{1}{q_o K_L} + \frac{1}{q_o} C_e \quad (5)$$

The equilibrium data were analyzed using the linearized form the Langmuir adsorption isotherm Eq. (5).

Table 1: The isotherm constant for Ni(II) adsorption onto ash

Langmuir equation		
$q_0$	$k_i$	$r^2$
166.67	0.014	0.772
Freundlich parameter		
$k$	$n$	$r^2$
2.7	1.16	0.996

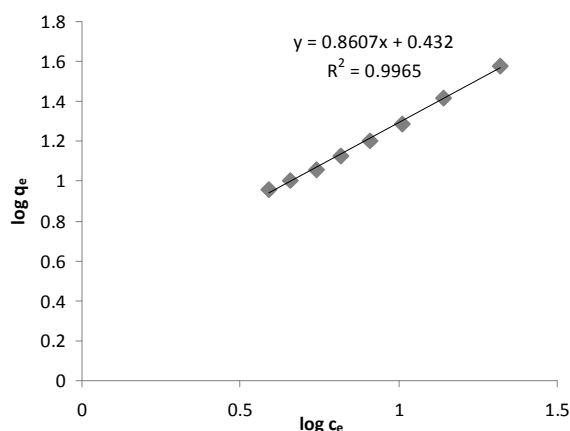


Fig. 12: Freundlich sorption isotherm for Ni(II) (the initial concentration, pH, volume of solution and contact time was 40 ppm, 3, 100 ml and 14 min respectively).

The Langmuir constants,  $K_L$  and monolayer sorption capacity,  $q_m$  were calculated from the slope and intercept of the plot between  $C_e/q_e$  and  $C_e$  (Fig.11). The results and equations are indicated in Fig.11 and Table 1. As can be seen, the slope of line is negative, so this equation is not suitable for these data.

**The Freundlich Isotherm Model:** While Langmuir isotherm assumes that enthalpy of adsorption is independent of the amount adsorbed, the empirical Freundlich equation, based on sorption on heterogeneous surface, can be derived assuming a logarithmic decrease in the enthalpy of adsorption with the increase in the fraction of occupied sites. The Freundlich equation is purely empirical based on sorption on heterogeneous surface and is given by:

$$q_e = K_F (C_e)^{1/n} \quad (6)$$

$K_F$  and  $(1/n)$  are the Freundlich constant and adsorption intensity, respectively. Equilibrium constants evaluated from the intercept and the

slope, respectively, of the linear plot of  $\log q_e$  versus  $\log C_e$  based on experimental data. The Freundlich equation can be linearized in logarithmic form for the determination of the Freundlich constants as shown below:

$$\log(q_e) = \log(K_F) + \frac{1}{n} \log(C_e) \quad (7)$$

The slope and the intercept correspond to  $(1/n)$  and  $K_F$ , respectively. It was revealed that the plot of  $\log q_e$  and  $\log C_e$  yields a straight line (Fig.12). The results are indicated in Table1.

## CONCLUSIONS

The PPY/ PPA composite showed considerable potential for the removal of Ni (II) from aqueous solutions. The optimum conditions of sorption were found to be: a sorbent dose of 0.4 g in 100 ml of Ni(II) contact time of 14 min, pH 3 for the Ni(II). The results gained from this study were well described by the theoretical Freundlich. The kinetic data indicated that the adsorption process was controlled by pseudo-second-order equation.

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