Pre-concentration of some heavy metal ions with AlO–HQ and AlO–PHQ and their studies by FTIR and spectroscopy

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Abstract Immobilization of HQ and PHQ on the surface of alumina, Al₂O₃ (referred to it by AlO) as an application for cation exchange of Al(III) in solution, using them for the extraction of some heavy metal ions from their solutions and calculating their capacity was studied. Immobilization of HQ or PHQ on Al₂O₃ was carried out and used for the extraction of some metal ions such as Fe(III), Cu(II), Co(II) and Ni(II) from its solution. Spectra of substrate (AlO–HQ, AlO–PHQ or AlO–Cu) at universal buffer solution (pH 7.2 ± 0.1) were recorded, solution of metal ion was added after contact time, filtered, and the solid precipitate was dissolved in pure ethanol, a peak at 305 nm was observed corresponding to HQ–Al(III). On addition of Cu(II) ion solution to AlO–HQ, a peak at 380 nm appeared corresponding to HQ–Cu(II), the peak at 305 nm decreased until it disappeared. The capacity of AlO–HQ was 100 mmol/g, whereas on addition of Fe(III) ion solution to AlO–HQ, a peak at 380 nm appeared corresponding to HQ–Cu(II), the peak at 305 nm decreased until it disappeared. The capacity of AlO–HQ was 100 mmol/g, whereas on addition of Fe(III) ion solution to AlO–HQ, a peak at 380 nm appeared corresponding to HQ–Cu(II), the peak at 305 nm decreased until it disappeared. The capacity of AlO–HQ for Fe(III) ion was 100 mmol/g. On addition of Ni(II) and Co(II), the capacity of AlO–HQ for the extraction of Ni(II) and Co(II) are 62.5 and 150 mmol/g, respectively. In the same conditions, and in the same way, Uv–visible absorption spectra of some metal ions extracted by AlO–PHQ were determined and the capacity of these metals was...
Tris(8-hydroxyquinoline) aluminum is presently considered as one of the most reliable emitting materials because of its excellent stability and luminescent properties. In the molecules, the quinolines function as bidentate ligands and bind to the aluminum atom through both nitrogen and oxygen atoms, and the coordination number is 6. The optical transition responsible for photoluminescence in AlQ3 is centered on the organic ligand. As the covalent nature of the metal–nitrogen bond is decreased, the emission shifts to shorter wavelength (Chen and Shi, 1998).

In recent years, the flat-panel display industry has grown tremendously due mostly to the success of liquid–crystal displays (LCD). However during this period, the technology of organic light-emitting diodes (OLED's) has advanced so that it now can compete directly with LCD for high-information content display applications (Amy, 2004). The more important it now can compete directly with LCD for high-information OLED's were computed and compared through computational calculations of both electronic and chemical reactivity descriptors. All the geometries were distorted tetrahedral, where both angles and bond length increased in the order Be$^{2+} <$ Zn$^{2+} <$ Cd$^{2+}$. The molecular orbital analysis reveals that the frontier orbitals are delocalized and preserve largely the electronic structure of the individual 8-hydroxyquinoline ligand, being practically unaffected by both the amidopyridine group and the metal cation. The more reactive sites were found in atoms belonging to the lowest unoccupied molecular orbital, which are in para-position to the quinoline nitrogen (Nu-Zarur and Vivas-Reyes, 2008).

Tris(8-hydroxyquinoline) aluminum (AlQ3) based transistor applications were very limited by relatively low currents and high operational voltages (Zorba and Gao, 2005). Chang-Hee Hong (Uthirakumar et al., 2008), performed an investigation of amorphous tris(8-hydroxyquinoline) aluminum (AlQ3) molecules transferred into α-phase crystalline nanomaterials by incorporating lower weight percentage of crystalline zinc oxide (ZnO) nanoparticles. Various shapes of hybrid AlQ3–ZnO nanomaterials were synthesized from a one-step solution method at relatively very low temperature. They examined the effect of temperatures and the amount of incorporated ZnO nanoparticles on the evolution of morphologies of hybrid nanomaterials by using field emission scanning electron microscopy. Photo Luminescence (PL) spectra of hybrid nanomaterials containing 15% and 25% of ZnO showed a three and five-fold enhancement in PL intensity, respectively, over the pure AlQ3 molecules. They attributed the achievement of higher PL intensity of AlQ3–ZnO hybrids to the incorporation of crystalline ZnO

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nanoparticles. This incorporation allowed for energy transfer from ZnO particles and the occurrence of caging effect of ZnO nanoparticles thus avoiding excimer formation between AlQ3 molecules in the excited state. A blue-shift in the PL emission of the hybrid nanomaterials resulted, with respect to ZnO content, due to the quantum confinement effect of the ZnO nanoparticles.

AlQ3 derivatives such as Al(Et)2Q (Yamaguchi et al., 2002), Al(Et)Q2 (Yamamoto and Iijima, 2004) and Al(Q)2(OH) (Leung et al., 2000) can be incorporated into polymers containing OH side groups by condensation reactions. High reactivity of the former and low solubility of the latter mentioned compounds can be overcome by replacing the ethyl or hydroxide group with long alkoxy groups. Furthermore, it has recently been proposed that the aluminum–nitrogen bond strength can be used as a parameter for tuning the complex emission wavelength (Chen and Shi, 1998), alternatively it may be possible to alter the emission wavelength to some extent using various alkoxy groups. As a contribution to the latest development of this interesting field, the electrochemical activity of poly(8-hydroxyquinoline) (PHQ) and its copper ion chelators in different solutions by the use of differential pulse polarographic (DPP) technique was studied (Kamal et al., 2002). The procedure was successfully used to determine analytically the PHQ matrix. The determination was successfully applied in the presence of a variety of anions, cations and in an insulating poly(vinyl alcohol) (PVA) matrix.

Kamal and co-workers (Ahmed et al., 2003), have prepared spectro analytical solutions and using several spectroscopic techniques, characterized coordination polymer based on poly(8-hydroxyquinoline) (PHQ) and its copper ion chelators in different solutions by the use of differential pulse polarographic (DPP) technique was studied (Kamal et al., 2002). The procedure was successfully used to determine analytically the PHQ matrix. The procedure was successfully applied in the presence of a variety of anions, cations and in an insulating poly(vinyl alcohol) (PVA) matrix.

2. Experimental

2.1. Materials

8-Hydroxyquinoline (HQ) was obtained from Aldrich (Chemical Co., US). Poly(8-hydroxyquinoline), PHQ, was prepared based on the homo-oxidative polymerization technique of the corresponding monomer, 8-HQ (Kamal et al., 2002). Metal ions such as: Co(II), Ni(II), Zn(II), Fe(III), Cu(II), Mg(II), Cr(III), Mn(II) and Mo(VI) ions via cation-exchange mechanism has been studied (Kamal et al., 2009).

2.2. Immobilization of 8-hydroxyquinoline (HQ) and Its polymer (PHQ)

Weigh about 1 g of 8-hydroxyquinoline (HQ), dissolve it in a small amount of pure ethanol (5 mL), add this solution to 10 g of aluminum oxide and mix well. Add to this swelling mixture 50 mL double distilled water in a 250 mL conical flask and stir for about 4 h. Filter this solution, wash the precipitate by double distilled water and leave it for drying at room temperature. On the third day, collect the solid yield in a closed bottle. HQ was immobilized on aluminum oxide (AlO–HQ). FTIR and Uv-spectroscopic measurements were carried out on this solid.

Immobilization of poly(8-hydroxyquinoline) on Alumina (AlO–PHQ) used for extraction of Ni(II) from solution. 15g of the nickel alloy sample was weighed, put in a 250 mL beaker, 50 mL concentrated nitric acid was added, and warmed with gentle heating on a hot plate for 1 h, the solution was cooled and left for three days. Weight of the residual alloy was 8.47 g, the weight of the dissolved metals material was 6.53 g, the solution was diluted to 100 mL by distilled water in a volumetric flask.

A perkin-Elmer Lambda 35 spectrophotometer with 1.0 cm quartz cells (scan speed, 8 nm/s) was used for spectrophotometric measurements. The pH measurements were made using a Jenway 3305 pH-meter accurate to ±0.01 pH unit with glass calomel electrode assembly. The pH-meter was standardized against pH values 4.0 and 10.0 using buffers prepared by dissolving buffer capsules in definite amounts of secondary deionized water. Magnetic stirrer Jenway 1000 was used for stirring the solutions. FTIR spectroscopic measurements on KBr pellets were scanned on a Shimadzu (type 470) spectrometer.

3. Results and discussion

3.1. FTIR of immobilized HQ on alumina (AlO)

After preparation of the immobilized HQ on alumina (AlO) to form coordinated aluminum oxide–(8-hydroxyquinoline) complex (AlO–HQ), the ability of this solid for extraction of some heavy metals was studied.

![Figure 1](https://via.placeholder.com/150)

**Figure 1** FTIR of, (a) alumina free AlO, (b) AlO–PHQ, (c) Co(II), and (d) AlO–PHQ–Co(II).
FTIR was used to prove the immobilization of HQ on the surface of AlO and Co(II) ion was extracted from the solution as shown in Fig. 1. The spectrum (a) of AlO was compared with the spectrum (b) of AlO–HQ, the absorption bands between 1400–1600 cm$^{-1}$ characteristic of HQ moiety prove the immobilization of HQ on the surface of AlO. The spectrum (c) for Co(II) ion has a main characteristic peak at 1640 cm$^{-1}$. The same peak appeared at spectrum (d) interrupted with the peaks of HQ proved that Co(II) coordinated with AlO–HQ to form AlO–HQ–Co(II).

3.2. Uv–visible spectroscopic studies of HQ and PHQ immobilized on AlO [AlO–HQ and AlO–PHQ]

3.2.1. Uv–visible absorption spectra of Cu(II) ion on AlO–HQ and on AlO–PHQ

Spectra of 20 mg of the solution containing AlO–HQ after different addition of Cu(II) ion solution with universal buffer (pH 7.2) was recorded as shown in Fig. 2. After contact time (1 min), the content was filtered, the solid precipitate was dissolved in 5 mL of pure ethanol and the spectra of that solution were recorded. On the first addition of Cu(II), a peak appeared at 305 nm corresponding to the peak of HQ–Al(III), which was produced from HQ immobilized on AlO, the very low concentration of Cu(II) ion still has no effect. While, after addition of larger concentration of Cu(II) ion, a new peak at 380 nm appeared corresponding to the peak of HQ–Cu(II) complex. The formation of this coordination complex indicates that the solid of HQ immobilized into AlO can extract the metal of Cu(II) ion from the solution to form AlO–HQ–Cu(II). Increases of the concentration of Cu(II) ion with increase of the formation of AlO–HQ–Cu(II) coordinated complex was observed, that could be confirmed by the increase of the absorbance peak at 380 nm, while the peak at 305 nm decreases due to the decrease of the HQ–Al(III) complex. This observation indicates that the Cu(II) ion has the ability to replace the Al(III) ion in the AlO–HQ chain by cation exchange mechanism.

The maximum absorbance of this peak as shown in Fig. 2f which corresponds to the added Cu(II) concentration of 2 mmol L$^{-1}$, while the peak at 305 nm completely disappeared. This Cu(II) ion concentration represents the capacity of AlO–HQ solid for extraction of Cu(II) ion from the solution by stripping HQ from AlO–HQ. This capacity was 100 mmol/g.

In the same way, and in the same conditions, spectra of 20 mg of AlO–PHQ after different addition of Cu(II) ion added at universal buffer solution (pH 7.2) were recorded as shown in Fig. 3.

The maximum absorbance of this peak as shown in Fig. 3 corresponds to the added Cu(II) concentration of 10 mmol L$^{-1}$. This Cu(II) ion concentration represents the capacity of AlO–PHQ solid for extraction of Cu(II) ion from the solution by stripping PHQ from AlO–PHQ. This capacity was 500 mmol/g.

Comparing the capacity of AlO–PHQ for the extraction of Cu(II) ion and the capacity of AlO–HQ, it was observed that the AlO–PHQ has the ability about five times greater than AlO–HQ. This means that, PHQ has the ability to immobilize on AlO and coordinated with Cu(II) ion more by five times compare to the ability of HQ to do that.

![Figure 2](image1.png)  
**Figure 2** Effect of different concentration of Cu(II) on 20 mg of AlO–HQ at universal buffer solution (pH 7.2). The concentration of Cu(II) are: [(a): 0.05, (b): 0.15, (c): 0.25, (d): 0.5, (e): 1.0, and (f): 2.0 mmol L$^{-1}$].

![Figure 3](image2.png)  
**Figure 3** Effect of different concentration of Cu(II) on 20 mg AlO–PHQ at universal buffer solution (pH 7.2). The concentration of Cu(II) are: [(a): 0.5, (b): 1, (c): 2, (d): 5, (e): 7, and (f): 10 mmol L$^{-1}$].
3.2.2. Uv–visible absorption spectra of Fe(III) ion on the immobilized substrate, AlO–HQ or AlO–PHQ

Spectra of different concentrations of Fe(III) ion on 20 mg of AlO–HQ at universal buffer solution (pH 7.2) were recorded as shown in Fig. 4. A solution of Fe(III) ion was added to the solution containing AlO–HQ, after contact time, the content was filtered, the solid precipitate was dissolved in 5 mL pure ethanol and the spectra of that solution was recorded. Two new peaks appeared at 580 and 455 nm corresponding to the formation of HQ–Fe(III) complex. The formation of this coordination complex indicates that the solid of HQ immobilized on AlO can extract the metal of Fe(III) ion from the solution to form AlO–HQ–Fe(III). Ethanol can dissolve the coordination complex HQ–Fe(III) and give the two appeared absorbance peaks at 580 and 455 nm.

Increases of the concentration of Fe(III) ion with increase in the formation of AlO–HQ–Fe(III) coordinated complex was observed, then the absorbance peaks increased. The maximum absorbance peak as shown in Fig. 4f corresponds to the added Fe(III) concentration of 2 mmol L$^{-1}$. This Fe(III) ion concentration represents the capacity of AlO–HQ solid for extraction of Fe(III) ion from the solution which was 100 mmol/g.

By the same way, and in the same conditions, Spectra of different concentrations of Fe(III) ion on 20 mg of AlO–PHQ at universal buffer solution (pH 7.2) were recorded as shown in Fig. 5. The maximum absorbance peak as shown in Fig. 5f which corresponds to the added Fe(III) concentration of 10 mmol L$^{-1}$. This Fe(III) ion concentration represents the capacity of AlO–PHQ solid for extraction of Fe(III) ion from the solution which was 500 mmol/g.

3.2.3. Linearity range of addition of Cu(II) or Fe(III) on the immobilized substrate, AlO–HQ or AlO–PHQ

Plotting of the concentration of Cu(II) ion added (mmol L$^{-1}$) against absorbance, as shown in Fig. 6, the linearity of this curve represents the detection limit range that can be used for applications; more than this concentration gives the logarithmic curve.

The range between (0.0–1.0 mmol L$^{-1}$) was the linear range ($r = 0.9914$) of addition of Cu(II) ion on AlO–HQ. While, the range between (0.2–2 mmol L$^{-1}$) was the linear range ($r = 0.993$) of addition of Cu(II) ion on AlO–PHQ.

Also, while plotting of the concentration of Fe(III) ion added (mmol L$^{-1}$) against absorbance, the range between (0.0–1.0 mmol L$^{-1}$) was the linear range ($r = 0.988$) of addition of Fe(III) ion on AlO–HQ while, the range between (0–2 mmol L$^{-1}$) was the linear range ($r = 0.974$) of addition of Fe(III) ion on AlO–PHQ.

This is the range that can be used for applications; more than this concentration gives the logarithmic curve until it reached 10 mmol L$^{-1}$.
3.3. Uv–visible absorption spectra of Ni(II) ion on AlO–HQ and on AlO–PHQ

Spectra of 20 mg of AlO–HQ after different addition of Ni(II) ion added with universal buffer solution (pH 7.2) were recorded as shown in Fig. 7. A peak at 305 nm corresponding to formation of HQ–Al(III) coordinated complex appeared, as seen in Fig. 6. The solution of Ni(II) ion was added to the solution containing AlO–HQ, after contact time, the content was filtered, the solid precipitate was dissolved in 5 mL pure ethanol and the spectra of that solution was recorded. On addition of individual different concentrations of Ni(II) ion, the absorption peak at 305 nm decreased, as shown in Fig. 7b–h. These observations could be attributed to cation exchange mechanism which proves that the Al(III) ion has the ability to replace Ni(II) ion in the HQ–Ni(II) chain when Al(III) was added to the solution of HQ–Ni(II). But Ni(II) ion does not have the ability to strip HQ which immobilized on AlO and forms the HQ–Al(III) coordinated complex. Then there was a decrease of the absorbance peak due to formation of metal–metal interchelation between [Al–Ni], then the coordination complex HQ–Al(III) decreased. The solid of HQ immobilized into AlO can extract the metal of Ni(II) ion from the solution to form the Ni–AlO–HQ interchelated complex. The maximum amount of Ni(II) that can be added to the solution containing 20 mg of AlO–HQ was 1.25 mmol L$^{-1}$, which causes the peak at 305 nm to nearly disappear. This concentration represents the capacity of AlO–HQ solid for extraction of Ni(II) ion, it was 62.5 mmol/g.

In the same way, and in the same conditions, the spectra of 20 mg of AlO–PHQ after different addition of Ni(II) ion added to universal buffer solution (pH 7.2) was recorded. The maximum amount of Ni(II) that can be extracted from this solution was 1.5 mmol L$^{-1}$, which represents the capacity of AlO–PHQ solid for extraction of Ni(II) ion, it was 75 mmol/g.

![Figure 6](image1.png)

**Figure 6** Plot of different concentration of Cu(II) or Fe(III) (mmol L$^{-1}$), at universal buffer solution (pH 7.2), on 20 mg of [AlO–HQ or AlO–PHQ] vs absorbance.

![Figure 7](image2.png)

**Figure 7** Effect of different concentration of Ni(II) on 20 mg of AlO–HQ at universal buffer solution (pH 7.2). The concentration of Ni(II) are: [(a): 0, (b): 0.05, (c): 0.15, (d): 0.25, (e): 0.5, (f): 0.75, (g): 1.0 and (h): 1.25 mmol L$^{-1}$].

![Figure 8](image3.png)

**Figure 8** Effect of different concentration of Co(II) on 20 mg of AlO–HQ at universal buffer solution (pH 7.2). The concentration of Co(II) ion are: [(a): 0.3, (b): 0.5, (c): 0.75, (d): 1.0, (e):1.5, (f): 2.0, and (g): 3.0 mmol L$^{-1}$].
3.4. Uv–visible absorption spectra of Co(II) ion on AlO–HQ and on AlO–PHQ

Spectra of solution containing 20 mg of AlO–HQ after different addition of Co(II) ion added at universal buffer solution (pH 7.2) was recorded as shown in Fig. 8. After contact time, the content was filtered, the solid precipitate was dissolved in 5 mL pure ethanol and the spectra of that solution was recorded. A peak at 380 nm corresponds to the formation of the HQ–Co(II) coordinated complex, as shown in Fig. 8a. On addition of individual different concentrations of Co(II) ion, the absorption peak at 380 nm decreased, as shown in Fig. 8b–g. These observations could be attributed to cation exchange mechanism which prove that Al(III) ion has the ability to replace Co(II) ion in the HQ–Co(II) chain when Al(III) was added to the solution of HQ–Co(II). But Co(II) ion can not stripp HQ which immobilized on AlO and forming HQ–Al(III) coordinated complex. Then the decrease of the absorbance peak due to formation of metal–metal interchelation between [Al–Co], then the coordination complex HQ–Al(III) was observed. So, the solid of HQ immobilized into AlO can extract the metal of Co(II) ion from the solution to form the Co–AlO–HQ interchelated complex.

The maximum amount of Co(II) that can be added to the solution containing 20 mg of AlO–HQ was 3 mmol L\(^{-1}\), which causes the two peaks at 305 and 380 nm to nearly disappear, as shown in Fig. 8g. This concentration represents the capacity of AlO–HQ solid for the extraction of Co(II) ion, it was 150 mmol/g.

In the same way and in the same conditions, the spectra of 20 mg of AlO–PHQ after different addition of Co(II) ion added to universal buffer solution (pH 7.2) were recorded. The maximum amount of Co(II) that can be extracted from the solution containing 20 mg of AlO–PHQ was 5 mmol L\(^{-1}\). This concentration represents the capacity of AlO–PHQ solid for extraction of Co(II) ion, it was 250 mmol/g.

3.5. Linearity range of addition of Ni(II) or Co(II) on the immobilized substrate, AlO–HQ or AlO–PHQ

Plotting of the concentration of Ni(II) ion added (mmol L\(^{-1}\)) against absorbance, as shown in Fig. 9, the linearity of this curve represents the detection limit range. The range between (0.0–1.25 mmol L\(^{-1}\)) was the linear range \(r = 0.9861\) of addition of Ni(II) ion on AlO–HQ. Also, the range between (0–1.25 mmol L\(^{-1}\)) was the linear range \(r = 0.988\) of addition of Ni(II) ion on AlO–PHQ.

While, plotting of the concentration of Co(II) ion added (mmol L\(^{-1}\)) against absorbance as shown in Fig. 9, the range between (0.0–2 mmol L\(^{-1}\)) was the linear range \(r = 0.9767\) of addition of Co(III) ion on AlO–HQ. The range between (0.0–2 mmol L\(^{-1}\)) was the linear range \(r = 0.992\) of addition of Co(III) ion on AlO–PHQ. This is the range that can be used for applications; more than this concentration gives a logarithmic curve until stable values of low absorbance are obtained.

4. Extraction of Ni(II) ion from solution of alloy sample

Immobilization of PHQ on alumina (AlO–PHQ) can be used for the removal of some metal ions from solution in different fields. The solution of nickel alloy was prepared as explained in Section 2.3. The spectra of 20 mg of AlO–PHQ at universal buffer solution (pH 7.2) was recorded, as shown in Fig. 10. A peak at 305 nm corresponding to PHQ–Al(III) was obtained.

![Figure 9](image)

**Figure 9** Plot of different concentration of Co(II) or Ni(II) (mmol L\(^{-1}\)), at universal buffer solution (pH 7.2), on 20 mg of [AlO–HQ or AlO–PHQ] vs absorbance.

![Figure 10](image)

**Figure 10** (a) Twenty milligrams of AlO–PHQ at universal buffer solution (pH 7.2). (b) Solution a + 0.5 mL of solution of nickel nut, filtrate, and the ppt. was dissolved in pure ethanol, (c) filtrate of b.

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Capacity of different immobilized forms on alumina (AlO).</th>
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<tbody>
<tr>
<td>Substrate</td>
<td>Cu(II)</td>
</tr>
<tr>
<td>AlO–HQ</td>
<td>100</td>
</tr>
<tr>
<td>AlO–PHQ</td>
<td>500</td>
</tr>
</tbody>
</table>
A 0.5 mL from the solution of nickel alloy was added, filtered, the precipitate was dissolved in 5 mL pure ethanol and the spectra of that solution was recorded, as shown in Fig. 10b. It is observed that the peak at 305 nm decreased due to the formation of metal-metal intercalation as Ni-AlO-PHQ, as explained previously. Another peak appears at 380 nm corresponding to the PHQ-M coordinated complex due to any metal ions that may be present in the alloy of the nickel alloy material.

To ensure that the AlO-PHQ solid extracts all metal ions present in that solution, spectra of the filtrate was recorded as shown in Fig. 10c. The two peaks at 305 and 380 nm disappeared.

Atomic Absorption Spectroscopy (AAS) also determined the recovery of this extraction and it was found to be 88.63%. This observation evaluated that the solid of AlO-PHQ can extract metal ions such as Ni(II) and Cu(II) from its solution.

5. Conclusions

The surface of alumina [aluminum oxide, Al₂O₃] can be modified with organic function groups as 8-hydroxyquinoline (HQ) or its polymer, poly(8-hydroxyquinoline) (PHQ), and provide access stability. As an application of cation exchange of Al(III) in solution, immobilization of 8-hydroxyquinoline (HQ) or its polymer (PHQ) on the surface of alumina [Al₂O₃, in this study signed by AlO], increases the capacity for extraction of heavy metal ions from solutions. It was used for the extraction of some heavy metal ion such as: Fe(III), Cu(II), Co(II) and Ni(II) from its solutions in different fields as in Table 1.

References