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M. POLAŃSKI^{*}, P. DESZCZ^{*}, K. KOŁCZYK^{*}, P. ZABIŃSKI^{*}**STUDY OF GOLD, COPPER AND NICKEL ADSORPTION, FROM THEIR ACIDIC CHLORIDE SOLUTIONS,
ONTO ACTIVATED CARBON**

In this paper, a simple and effective method for gold recovery is described. The paper describes a way to recover gold onto activated carbon from a synthetic solution of gold(III) chloride. The method can also be used on nickel(II) as well as copper(II) chloride of where the metal ion ratios are comparable to the metal ratios found in some electronic waste.

With the use of activated carbon in the process of electrolyte purification it is possible to selectively remove gold in metallic form from the solution. XPS studies have confirmed that metallic gold is present on the carbon surface. A spectrophotometric method was used to determine the concentration of Au(III) in the solution. Different concentration of nickel(II) as well as copper(II) were investigated. In all cases, adsorption and reduction of Au(III) to the metallic form was observed.

Keywords: adsorption, gold(III) chloride ions, recovery, recycling.

1. Introduction

The recovery of gold from electronic waste is one of the most important topics in the field of recycling. The selective separation of gold, from electronic waste is very difficult due to the low concentration of this metal in comparison with the accompanying metals. It is worth noting that gold recovery from electronic waste is a more complex process in comparison with its production from most of primary sources [1]. The recovery of gold from wastes can be achieved using several methods, for example by cementation using magnesium, copper, iron, zinc or aluminum [2]. Karavasteva has also shown, that the rate of gold recovery by cementation can be ranked as follow Cu>Zn>Mg>Fe>Al. It should be noted, that metal consumption during the cementation process is significant, especially for zinc in an acidic media. Another method often used for gold recovery is reduction. For this purpose iron(II) chloride [3], L-ascorbic acid [4], hydrazine [5], sulfur(IV) [6], HSO₃⁻ ions [7] or H₂O₂ [8] can be applied. In most of these processes, a single element system was investigated, and the behavior of other accompanying metals in the electronic waste is unknown.

Bizzo et al. [9] reviewed 13 papers dealing with composition of personal computers mother boards (PCBs). They have shown, that the concentration of gold in this type of electronic waste, varies from 27 to 1120 ppm where, for example, the concentration of other metals ranged between 12.5-28.7% of Cu, 1.7-7.06% of Al, 0.6-4.24 % of Fe, 0.26-2% of Ni, 0.08-2.7

of Zn%. In addition silver, lead and tin are present in the solder. The process of recycling of metal from electronic scrap using plasma furnace was also investigated by Jarosz et al. [10]. As evident, the recovery of gold from polymetallic sources using hydrometallurgical processes is a complex problem. This problem has been, at least, partially solved, and the recovery of gold from electronic waste is subject to ongoing work. However, we would like to shed light on new, probably not applied before method for gold recovery from such a waste.

Based on previous work which focused on gold recovery from diluted aqua solutions, using activated carbons, where no other metals were presents in the solution [11-13], new experiments were carried out with the application of idealized solutions comparable with e-waste liquor. These experiments have clearly shown, that the gold(III) ions present in the solution were adsorbed on different types of activated carbons, and reduced to metallic form. The results suggest that activated carbon can be used as a selective agent for gold recovery from e-waste, where due to adsorption and reduction, gold in metallic form can be removed. This adsorption is a well-known principle that was applied at the turn of the 19th/20th century on an industrial scale in Australia. E.g. the recovery of gold from chlorination leach liquors was patented by Davis in 1880 and the process was used at the Mont Morgan mine in Queensland (Australia). 11 years later the process was used to extract gold from primary sources.

This paper reports the results of the selective removal of gold from diluted aqueous solution using activated carbon Norit

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GF 40. The solutions used in experiments also contained Cu(II), and Ni(II) ions. The concentrations of each element (Au, Cu and Ni) was comparable to the concentration in PCBs wastes.

2. Experimental procedure

In all experiments, commercially available activated carbon, Norit GF 40 (AC) in non-modified form was used. Gold(III) chloride complex was obtained according to the methodology described in a previous paper [14].

The measurements of the concentration of gold(III) chloride complex ions after adsorption experiments were carried out using spectrophotometry (Shimadzu, model PC 2501, Japan). It is well known, that gold(III) chloride complex, has three peaks in the UV-Vis range. Unfortunately, when Cu(II) and Ni(II) are present in the solution, spectrophotometry cannot be used directly. Therefore, to determine the concentration of trace amounts of gold(III) in the solution, selective extraction was performed according to Marzenko protocol [15]. For this purpose, rhodamine B (Avantor Performance Materials) and benzene (Avantor Performance Materials) were used.

The appropriate amount of rhodamin B was dissolved in 1 M HCl to obtain a 0.04% (w/v) solution. To determine the concentration of Au(III), a 3 mL of analyzed sample was mixed with 1 mL of rhodamine B 0.04% solution, then 8 mL of benzene was added and shaken by hand for c.a. 1 minute. After this time the sample was left for phase separation. Next, the benzene phase was taken and analyzed using spectrophotometry in a 1 cm quartz cuvette. As a reference (blank) pure benzene was used.

All ion separation experiments were performed in a sealed glass batch reactor to prevent water evaporation. All experiments were carried out at 30°C. In all cases, the volume of solution used was 100 mL. The glass reactors were thermostatic and were shaken using an Elpin+ shaker.

The concentration of Ni(II) and Cu(II) (chloride salts, purchase from Avantor Performance Materials) was determined directly from aqua solutions using spectrophotometry. In this case, as a reference (blank) deionized water was used. It should be noted that the UV-Vis spectra of Cu(II) were taken using 0.2 cm quartz cuvette. To control the pH of the solution, hydrochloric acids (0.1 M) was used (Avantor Performance Materials).

The sample of activated carbon with deposited gold taken for XRD analysis was dried at 60°C for 1 day. Then, the pellet of activated carbon was pulverized using agate mortar and pestle. The powder was analyzed using rotating sample holder. XRD analysis was performed using Rigaku Miniflex II desktop X-Ray diffractometer. Registered XRD data were analyzed using PDXL software v. 1.7.0.0 (Rigaku Corporation).

The XPS analysis of the adsorbent after sorption experiments was carried out using SES R4000 equipped with hemispherical analyzer.

3. Results

3.1. Determination of molar absorption coefficient for Au(III)-rhodamin B complex in benzene

In the first step, the molar absorption coefficient for gold(III) chloride complex ions in benzene was determined. The results obtained are shown in Fig. 1. As the reference during the spectrophotometric measurements pure benzene was used.

According to Lambert-Beer law, the molar absorption coefficients were determined

$$Abs = \varepsilon_{Au(III)} \cdot l \cdot [Au(III)] \quad (1)$$

where

Abs – absorbance level

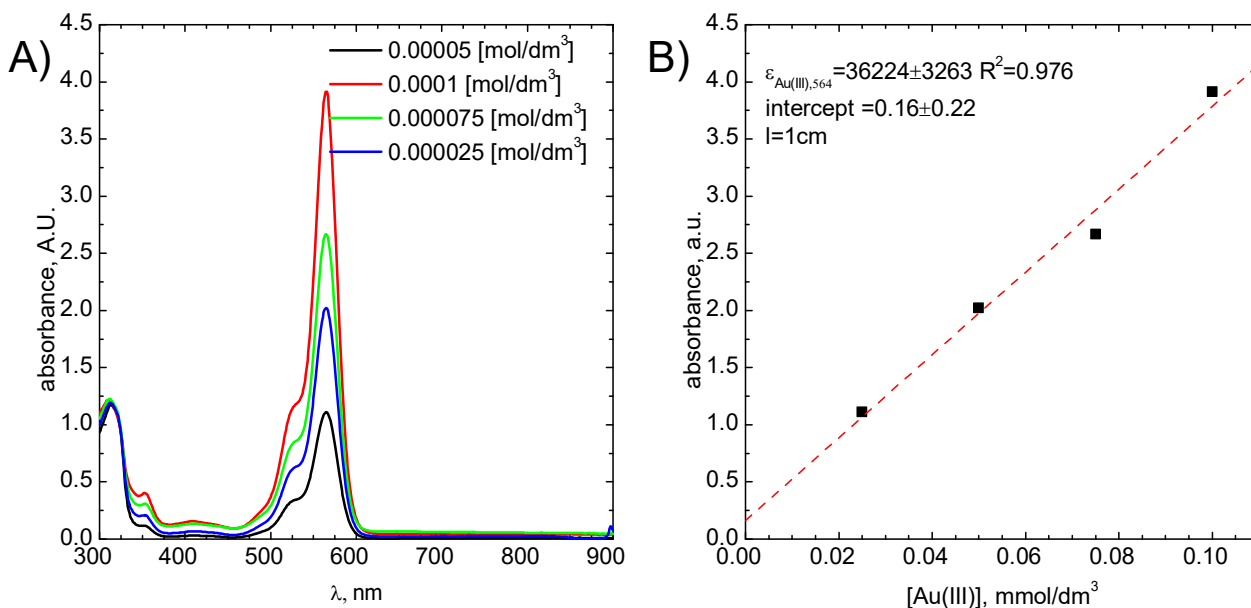


Fig. 1. A) UV-Vis spectra of Rhodamin B-Au(III) complex, B) determination of molar absorption coefficient of Rhodamin B-Au(III) complex

ε_x – molar absorption coefficient of Au(III) compound, $\text{cm}^{-1} \cdot \text{mol}^{-1} \cdot \text{dm}^3$

l – path length, cm

$[\text{Au(III)}]$ – the concentration of Au(III), mol/dm^3

It is possible to determine the molar absorption coefficient of gold(III)-rhodamin B complex compound by taking slope of Abs v.s. $[\text{Au(III)}]$ curve (see Fig. 1B). The maximum of absorbance is observed at the wavelength equal to 564 nm. Due to this it is also possible to determine the concentration of gold(III)-rhodamin B complex in unknown sample.

In Fig. 1B the slope did not pass through the origin of the coordination system. The intercept is equal to $0.16 \pm 0.22 \text{ cm}^{-1} \cdot \text{mol}^{-1} \cdot \text{dm}^3$ and the slope is equal to $36223 \pm 3263 \text{ cm}^{-1} \cdot \text{mol}^{-1} \cdot \text{dm}^3$. In fact the slope is the molar absorption coefficient for Au(III)-rhodamin B complex. The apparent value of intercept is directly related to solubility of water in benzene. This results in the solubility of some amounts of rhodamine B which has maximum absorption band located at the same wavelength as the Au(III) rhodamine B complex. The results were used to calculate the concentration of Au(III) after the adsorption process.

3.2. Determination of molar absorption coefficient for Ni(II)

Fig. 2A, shows the UV-Vis spectra of Ni^{2+} ions containing aqueous solutions. It can be seen, that nickel has 3 absorption bands located at 394 nm, 655 nm and 722 nm. To determine the molar absorption coefficient, a graphical method similar to that for gold-rhodamin B complex was applied. The result is shown in Fig. 2B.

The first peak located at wavelength 394 nm exhibits the highest molar absorption coefficient and is equal to $4.97 \pm 0.05 \text{ cm}^{-1} \cdot \text{mol}^{-1} \cdot \text{dm}^3$.

3.3. Determination of molar absorption coefficient for Cu(II)

Fig. 3A, shows the UV-Vis spectra of Cu^{2+} ions containing aqueous solutions at various concentrations. To determine the molar absorption coefficient, the graphical method as before was applied. The result is shown in Fig. 3B.

As it is well known, Cu^{2+} ions absorb light at the wavelength equal to 820 nm, and determined molar absorption coefficient is equal to $2.14 \pm 0.1105 \text{ cm}^{-1} \cdot \text{mol}^{-1} \cdot \text{dm}^3$. In case of Cu(II) cations, the UV-Vis absorption spectrum is independent to type of anions (chloride or sulfate) [16].

3.4. Gold recovery from Au(III)-Ni(II) system using activated carbon

Table 1 shows the composition of solutions used for gold recovery experiments. The $\Delta[\text{Ni(II)}]$ was calculated as the difference between initial concentration indexed as “0” and equilibrium concentration (after 7 days) indexed as “r”. Gold concentration in all experiments was the same and similar amounts of activated carbon ($0.25 \pm 0.0009 \text{ g}$) were used. Detailed information are shown in Table 1

TABLE 1

The composition of the solutions, prepared for Au(III) extraction in Au(III)-Ni(II) system

$[\text{Ni(II)}]_0, \text{ M}$	$[\text{Ni(II)}]_r, \text{ M}$	$\Delta[\text{Ni(II)}], \text{ M}$	$[\text{Au(III)}], \text{ M}$	$[\text{AC}], \text{ g}$
1.01×10^{-1}	1.0×10^{-1}	1.41×10^{-3}	1×10^{-4}	0.2499
7.98×10^{-2}	7.98×10^{-2}	0	1×10^{-4}	0.2501
5.87×10^{-2}	5.96×10^{-2}	-9.39×10^{-4}	1×10^{-4}	0.2491
3.85×10^{-2}	3.99×10^{-2}	-1.41×10^{-3}	1×10^{-4}	0.2509
1.88×10^{-2}	2.11×10^{-2}	-2.35×10^{-3}	1×10^{-4}	0.2492

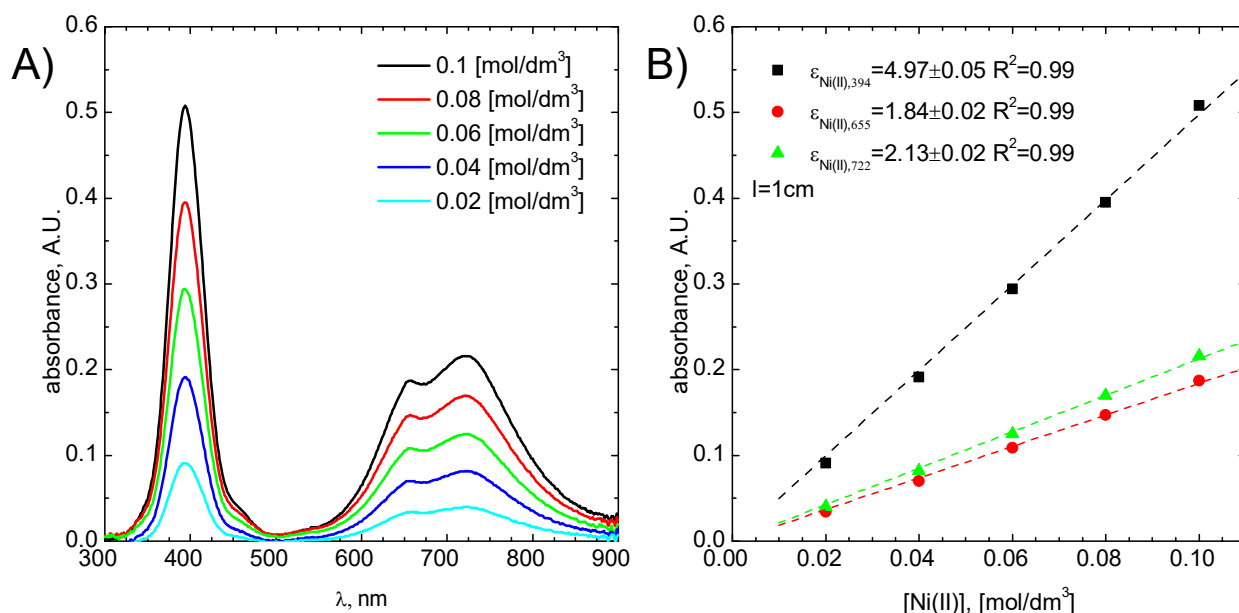


Fig. 2. A) UV-VIS spectra of Ni^{2+} containing aqueous solutions, $\text{pH} = 1$, $\text{Cl}^- = 0.1$, B) graphical determination of molar absorption coefficient of Ni^{2+}

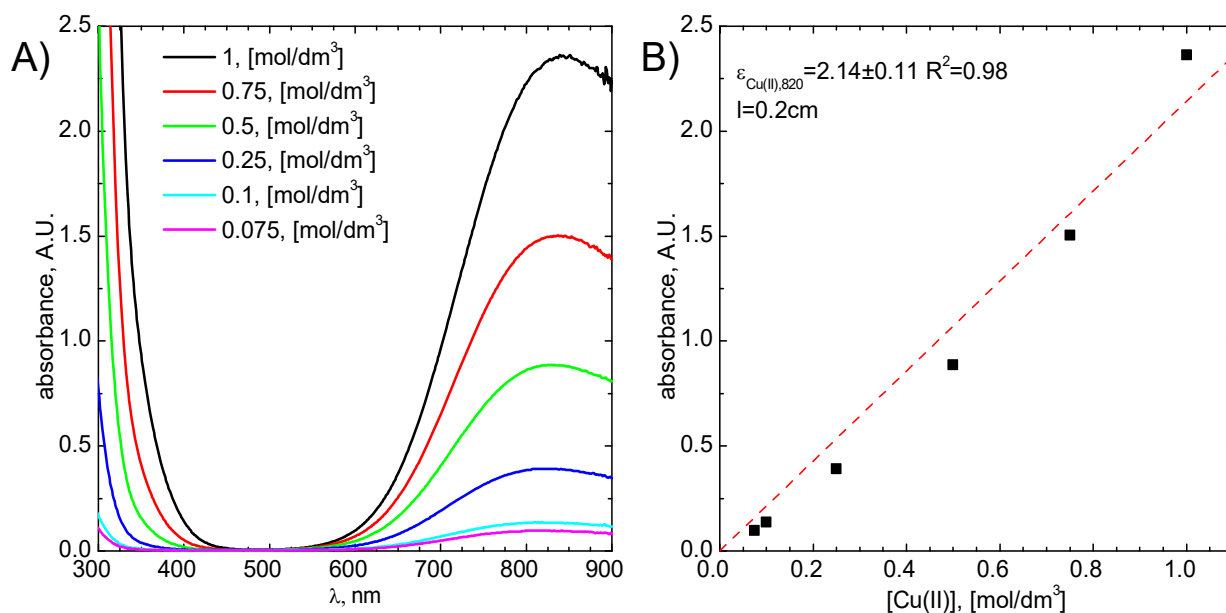


Fig. 3. A) UV-VIS spectra of Cu²⁺ containing aqueous solutions, pH = 1, Cl⁻ = 0.1, B) graphical determination of molar absorption coefficient

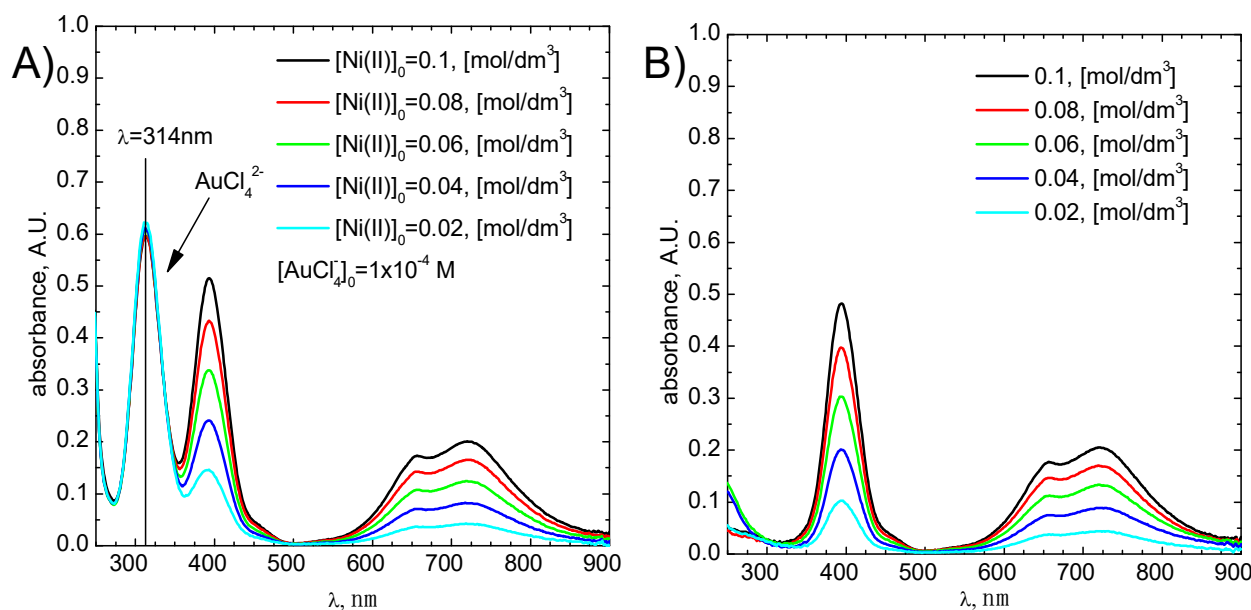


Fig. 4. A) UV-Vis spectra of aqueous solution containing Au(III) chloride complex ions, and different initial concentrations of Ni²⁺ before adsorption, B) UV-Vis spectra of aqueous solution after adsorption (7 days)

In Fig. 4A the UV-Vis spectra of the tested solutions are shown. The solutions contain, constant amounts of Au(III) chloride complex ions equal to 1×10^{-4} M, and different initial concentration of Ni²⁺ ions.

Gold(III) chloride complex ions absorb light in the range of UV-Vis, and have three absorption bands. The first band is located at wavelength 226 nm, in this case is overlapping with nickel(II) spectrum. The second one is located at the wavelength 314 nm where molar absorption coefficient is equal to $5600 \text{ cm}^{-1} \cdot \text{mol}^{-1} \cdot \text{dm}^3$ [17] and was used to determine the concentration of Au(III). The third one is located at the wavelength ~ 390 nm, however in this case the molar absorption coefficient is very low, and can be used only for concentrated samples. As it

can be seen, the peak observed at 314 nm wavelength, disappear after 7 days (see Fig. 4B). It is also observed that the absorbance level of Ni²⁺ ions remained constant (no changes of absorbance level at the wavelength of 394 nm). This suggest, that gold(III) ions were adsorbed selectively. Microscopic observation, has showed the presence of a brown deposit at the surface of the activated carbon (see Fig. 5).

Thanks to the XPS analysis it is possible to determine the electronic state of each element in the system. The results are shown in Fig. 6.

As it can be seen (Fig. 6A) at the surface of AC, nickel, gold as well as chloride elements are present. Detailed analysis of Au_{4f} has shown, that gold is present in the form of metal (82%)



Fig. 5. Microscopic image of activated carbon after adsorption process

and in the form of Au(III) about 18%. Insignificant binding energy shift is observed for Au_{7/2} this in turn suggest that the Au particles are nanometric.

It has to be noted, that orbital levels except the s levels, give a doublet with the two possible states having different binding energies. This is known as spin-orbit splitting [18] and in Fig. 6B is noted with prime symbols.

In case of nickel (see Fig. 6C), the amount on the surface is low. Therefore the signal is weak. It can be calculated that about 57% of adsorbed nickel exist in the form of nickel chloride and c.a. 43% is in the form of Ni-O in the surrounding of strongly electronegative compounds. However, the signal is strongly noised, and the quality of this signal is poor. It is related to low concentration of Ni in the sample. This in turn confirms that the process of gold adsorption is selective in respect to Ni.

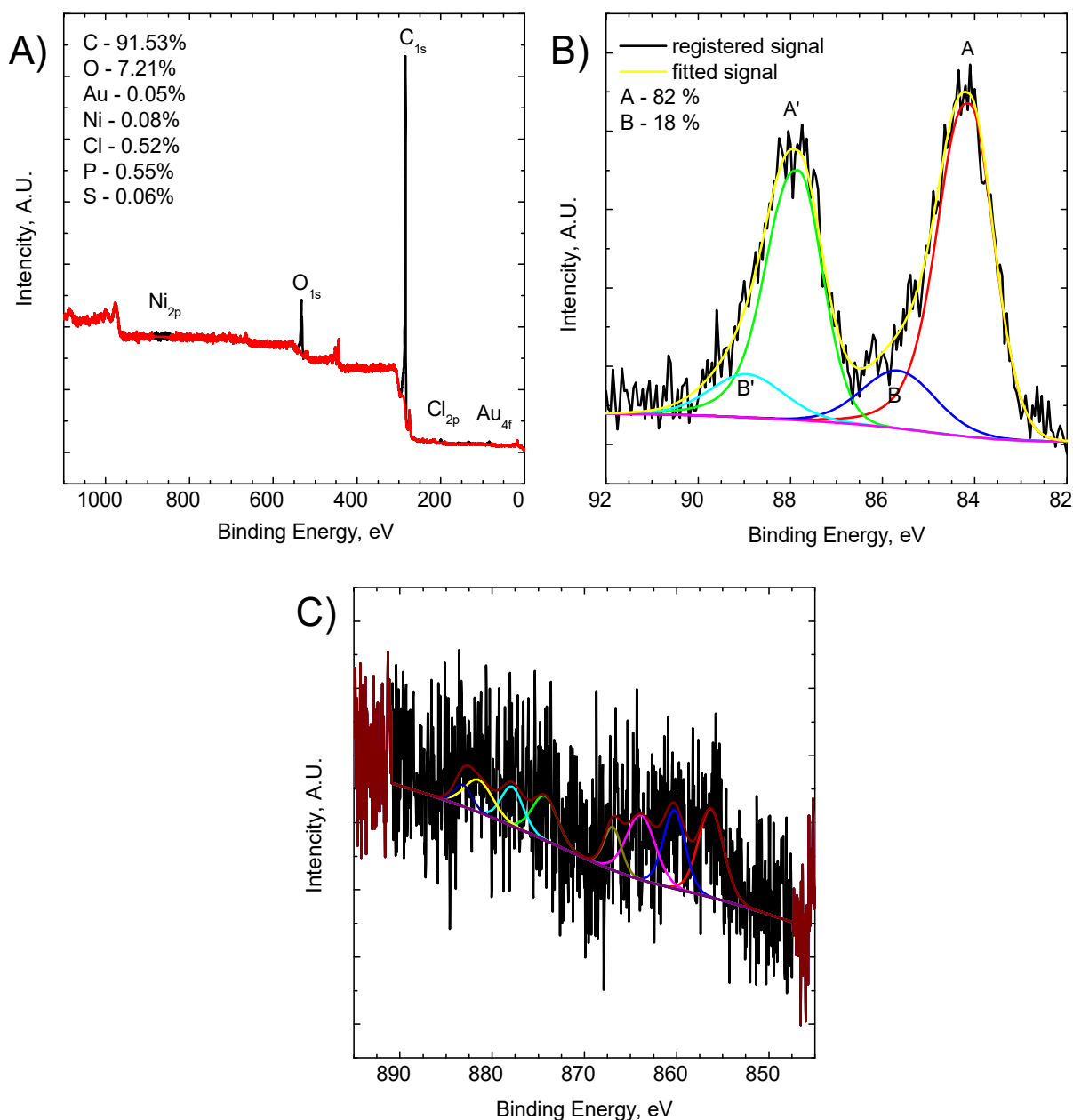


Fig. 6. XPS analysis of the sample after adsorption of gold from Ni-Cl-H₂O system, A) survey spectrum, B) electronic analysis of gold, C) electronic analysis of nickel

3.5. Gold recovery from Au(III)-Cu(II) system

In case of ions mixture Au(III) and Cu(II), direct Au(III) concentration observation is impossible using UV-Vis method. It is due to overlapping of Au(III) chloride complex ions spectra with Cu(II). Only changes of Cu(II) concentration can be observed using UV-Vis method. In

Table 2 composition of the solution prepared for Au(III) extraction, as well as changes in $\Delta[\text{Cu(II)}]$ concentration are shown. The changes of Cu(II) concentration can be calculated as follow:

$$\Delta[\text{Cu(II)}] = [\text{Cu(II)}]_0 - [\text{Cu(II)}]_r \quad (2)$$

TABLE 2

The composition of the solutions, prepared for Au(III) extraction

$[\text{Cu(II)}]_0, \text{ M}$	$[\text{Cu(II)}]_r, \text{ M}$	$\Delta[\text{Cu(II)}], \text{ M}$	$[\text{Au(III)}], \text{ M}$	$[\text{AC}], \text{ g}$
1.03	1.03	-7.01×10^{-3}	1×10^{-4}	0.25
7.15×10^{-1}	7.16×10^{-1}	-9.35×10^{-4}	1×10^{-4}	0.2511
4.25×10^{-1}	4.19×10^{-1}	6.07×10^{-3}	1×10^{-4}	0.2495
1.83×10^{-1}	1.79×10^{-1}	4.67×10^{-3}	1×10^{-4}	0.2499
6.92×10^{-2}	6.73×10^{-2}	1.87×10^{-3}	1×10^{-4}	0.2502
5.28×10^{-2}	5.23×10^{-2}	4.67×10^{-3}	1×10^{-4}	0.2505

It can be assumed, that the changes of amount of Cu(II) is smaller than the occurrence of applied method.

To observe the changes of Au(III) rhodamine B method was applied according to Marzenko protocol [15]. In

Fig. 7 UV-Vis spectra of organic phase after gold(III) extraction is shown. At the wavelength 559 nm a small peak can be observed.

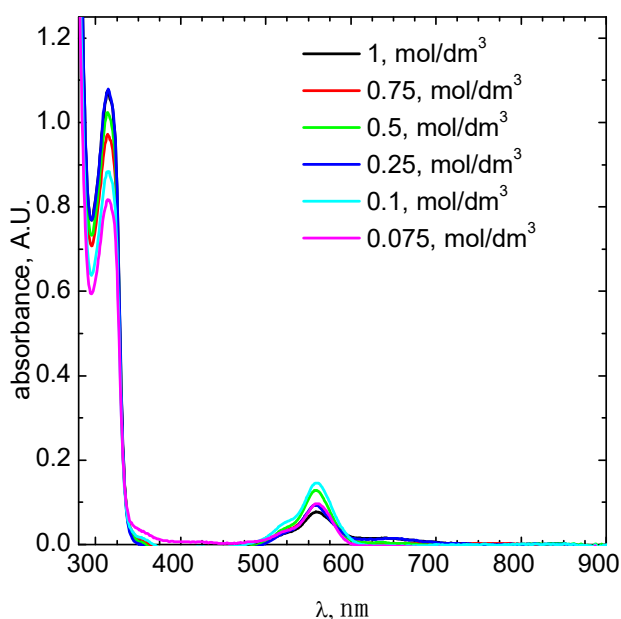


Fig. 7. UV-Vis spectra of organic phase after Au(III) extraction

As was shown earlier in section 3.1 during the graphical determination of molar absorption coefficient of Au(III)-Rhodamine

B complex, the intercept was equal to $0.16 \pm 0.22 \text{ cm}^{-1} \cdot \text{mol}^{-1} \cdot \text{dm}^3$. It was explained that this is an effect of water solubility in benzene. In case of results shown in Fig. 7 the absorbance level is fluctuating in the range from 0.078 to 0.146. So then it can be assumed that in the analyzed sample, the concentration of gold was below detection level. For the Rhodamine B method, the gold detection level is c.a. $2.5 \times 10^{-6} \text{ M}$ [15].

The surface of activated carbon after adsorption was analyzed using XRD method. Obtained result is shown in Fig. 8.

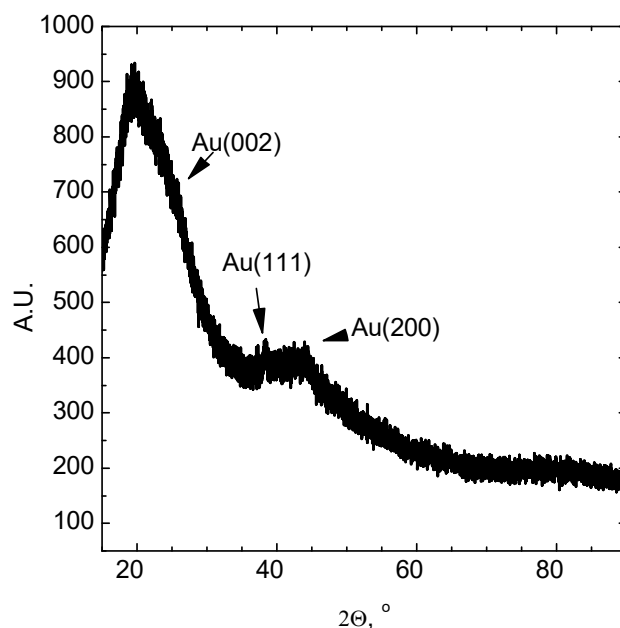


Fig. 8. XRD analysis of AC after adsorption of Au(III). Experimental conditions $[\text{Cu(II)}] = 0.07 \text{ M}$, $[\text{Au(III)}] = 2.5 \times 10^{-4} \text{ M}$, $[\text{AC}] = 2.5 \text{ g}$

Several peaks corresponding to metallic gold can be found. However to prove that there is only metallic gold present on the AC surface more sensitive methods have to be applied. Therefore a XPS analysis was also performed, and the result is shown in Fig. 9.

In (Fig. 9C), only the metallic form of the gold can be found. A slight shift of binding energy in the direction of higher values might be related to the small size of metallic particles in range of several nm. This results is, in turn, in good agreement to the XRD results, where peaks have low intensity and are broadened with is also related to small crystal size.

In respect to copper, it was found that it exists in three stages. The first one (Fig. 9B) denoted as A is related to Cu alloy – probably Au-Cu (about 13%). The second one labeled as B is related to metallic copper (about 76%) where third one labeled as C is related to CuCl_2 salt (about 11%).

3.6. Gold recovery from Au(III)-Ni(II)-Cu(II) system

Subsequently, gold recovery from Au(III)-Ni(II)-Cu(II) system was performed. For this purpose five different solutions were prepared. In each solution, concentration of gold was con-

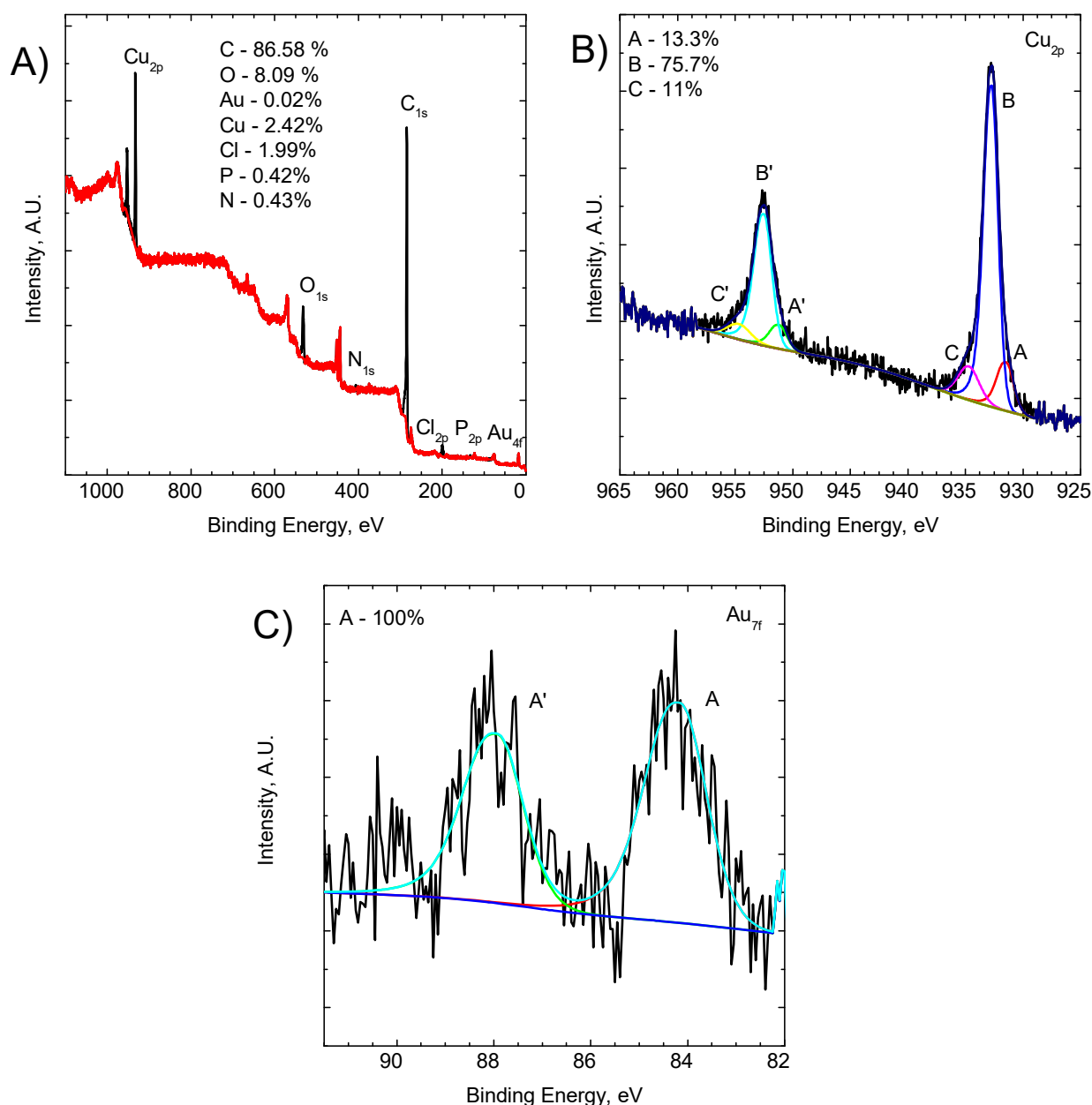


Fig. 9. XPS analysis of the sample after adsorption. A) survey spectrum, B) electronic analysis of copper, C) electronic analysis of gold. Experimental conditions $[\text{Cu(II)}] = 0.07\text{M}$, $[\text{Au(III)}] = 1 \times 10^{-4}\text{M}$, $[\text{AC}] = 0.25\text{g}$

stant and equal to $1 \times 10^{-4}\text{M}$, only the concentration of Ni(II) and Cu(II) were change, however the proportion was between Ni(II) and Cu(II) was also constant. Concentrations of each metal ions are shown in Table 3.

TABLE 3

The composition of the solutions, prepared for Au(III) extraction

$[\text{Ni(II)}]_0, \text{M}$	$[\text{Cu(II)}]_0, \text{M}$	$[\text{Au(III)}], \text{M}$	$[\text{AC}], \text{g}$
0.05	0.5	1×10^{-4}	0.2495
0.04	0.4	1×10^{-4}	0.2509
0.03	0.3	1×10^{-4}	0.2500
0.02	0.2	1×10^{-4}	0.2501
0.01	0.1	1×10^{-4}	0.2505

In Fig. 10A, UV-Vis spectrum of solutions before adsorption is shown. As it can be seen, only UV-Vis spectrum of Cu(II) can be distinguished.

In case of this system, to determine the concentration of Au(III) rhodamine B, the method used was as previously (see Fig. 10B). UV-Vis analysis have shown, that in the solution Au(III) ions are absent. This in turn suggest, that those ions are adsorbed on to the surface of the AC. To confirm this assumption, a XPS analysis was performed. The results are shown in Fig. 11. As it can be seen (Fig. 11A), gold, copper and chloride ions are adsorbed. A survey of the spectrum did not confirm the presence of nickel at the surface of activated carbon.

In case of gold, its fraction is equal to 0.01%, where c.a. 84% is in the form of metal and about 16% in the form of Au^{3+} . In respect to copper, about 10% is in the form of Au-Cu alloy,

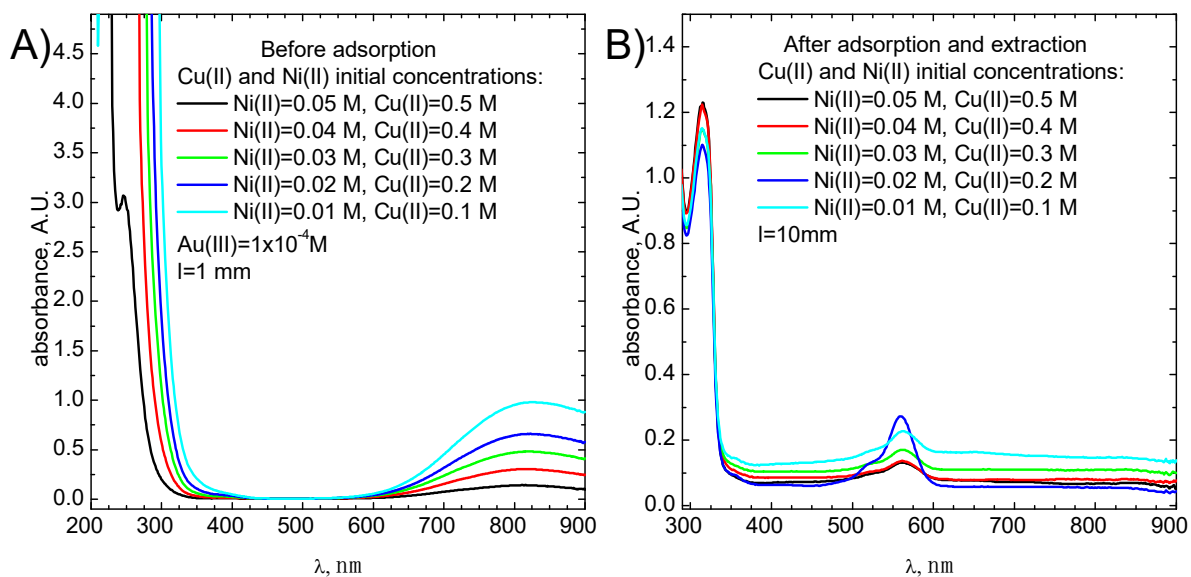


Fig. 10. UV-Vis spectrum of the solutions A) before adsorption and B) after adsorption end extraction using rhodamin B

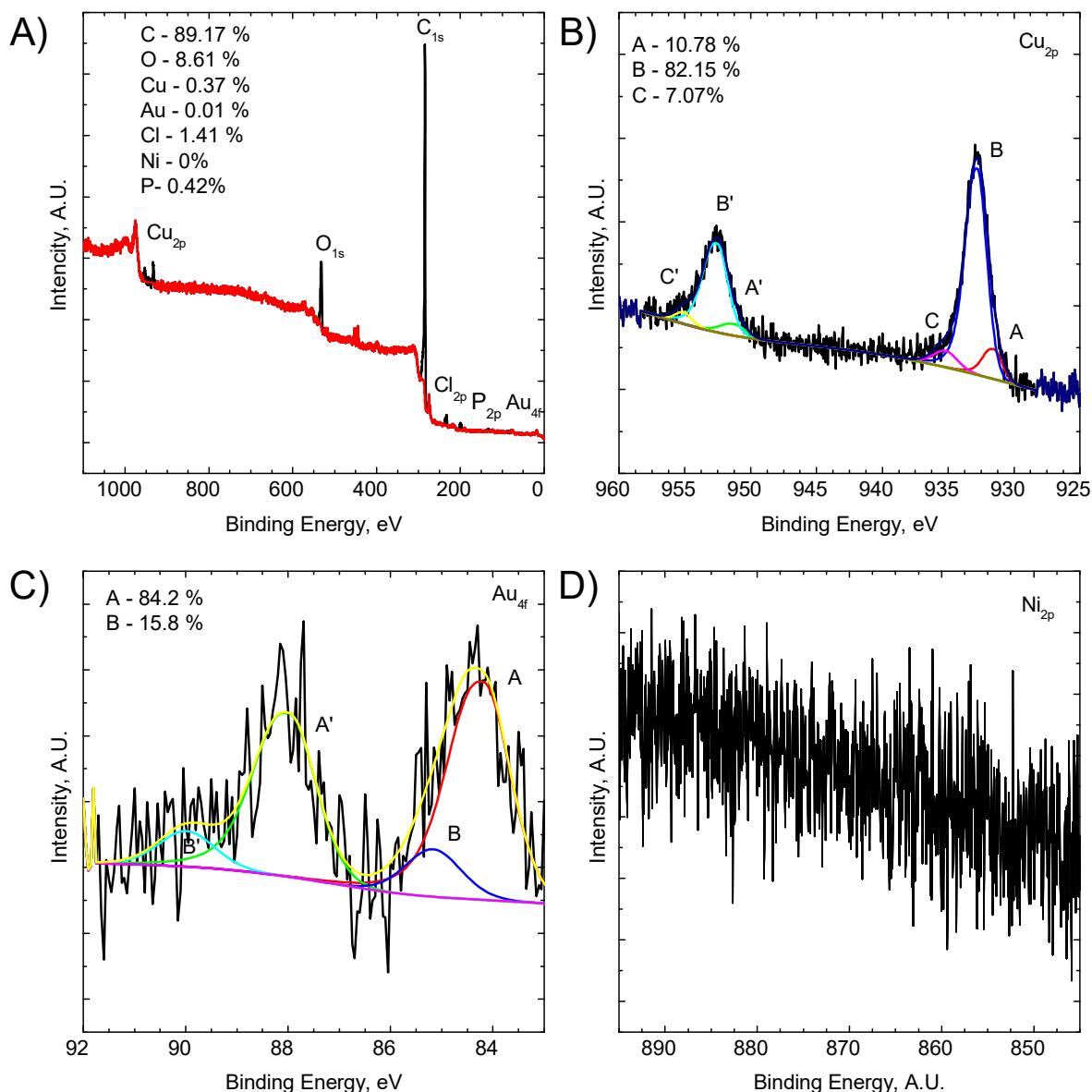


Fig. 11. XPS analysis of the sample after adsorption. A) survey spectrum, B) electronic analysis of copper, C) electronic analysis of gold, D) electronic analysis of nickel

82% in the form of metal and 7% in the form of Cu^{2+} salt, in this case probably CuCl_2 .

4. Discussion

The adsorption process of gold(III) chloride complex ions from solutions containing Cu(II), Ni(II) was investigated. In all investigated cases, changes in Cu(II) and Ni(II) concentration after adsorption is negligible. Adsorption efficiency of Au(III) is close to 100% and is related to the gold reduction to metallic form.

It was shown, that it is possible to selectively recover gold from the aqueous solutions containing Ni^{2+} and Cu^{2+} ions. In case of copper, some amounts of its metallic form occurs. However, in case of copper it is possible to selectively dissolve it, using sulphuric acid and oxygen as oxidant. The presented method is not expensive, therefore can be applied in the industrial scale, where amounts of generated solutions are significant. The method is also partially selective especially in respect to nickel, however in case of copper some of its amounts are also deposited on AC surface. In all experiments, gold was observed on the surface of the AC. Such an effect was also observed in our previous study [11-13], where it was confirmed that the presence of metallic gold at the external surface is related with diffusion control of the reduction reaction.

In case of Ni(II) adsorption, there is also many papers describing this process. However in strong acidic media it seems, that only trace amounts of Ni(II) are sorbed [19]. Similar results have been shown by Saif et al. [20].

Activated carbons can be used to recover gold during its recycling from very diluted solutions e.g. from recycling of electronic waste. The most important advantage of activated carbon application is its price and its high capacity in respect to gold.

5. Conclusions

Based on the experiments carried out following conclusions can be drawn.

Activated carbons can be used to recover gold from solutions similar to those encountered during the recycling of electronic waste. This method is selective in the recovery of gold from Au-Ni-Cu aqueous system. Adsorption efficiency of Au(III) is close to 100% and is related to the gold reduction to metallic form. Changes in Cu(II) and Ni(II) concentration after adsorption of gold is negligible.

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Solutions protected by patent application PL P.419685

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