# STABILITY OF TRIS-1, 10 – PHENANTHROLINE IRON (II) COMPLEX IN DIFFERENT COMPOSITES

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Received 04.02.3010.

Revised 20.04.2010.

Accepted 03.05.2010.

A part of this study was presented as a poster at the 8th Simposium „Novel Technologies and Economics Development", University of Niš, Faculty of Technology, Leskovac, October 23-24, 2009.

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**Abstract**

The composition of composites has a huge impact on the stability of tris-1, 10 – phenanthroline iron (II) complex during the determination of total iron. The subject of this work is determination the stability of tris-1, 10 – phenanthroline iron (II) complex in different composites. Composites with different concentration in which total iron was determined were alumina and zeolite. Stability of this complex was monitored in a time period of 0-60 min. The aim of this work is to determine the concentration of different composite samples and the time interval in which the stability of the complex is the highest. The spectrophotometric method was used for the determination. The tris-1, 10 – phenanthroline iron (II) complex with alumina is more stable at higher concentration of the samples. With zeolite, however, this complex is stable in a short time interval, regardless of the concentration of the sample. This conclusion indicates on increase of absorption, which is a consequence of impede complex formation and creating a new complex. For this reason, this method has only a limited application.

**Key words:** 1,10–phenanthroline iron (II) complex, composites, zeolite, stability alumina,

## Introduction

According to the theory of coordination, atoms or ions, particularly the atoms of transition elements, can be bound or coordinate in a certain number or they create a spatial distribution of atoms, ions or molecules, forming complex (coordination) compounds. Complex compounds consist of the central atom ion (builder of the complex) around which two or more ligands are coordinated. The number of ligands that are directly linked to the central ion in the complex is observed as the coordination number. It depends on the nature of the central atom, its electronic configuration and size, as well as coordination ability of the ligand [1,2,3,].

Polyatomic ligands can be bound to the central ion with one or more atoms, therefore we can distingush monodentate, bidentate, three-dentate or polydentate ligands. Coordination number can be 2, 3, 4, 5, 6, 7 and 8, but the most common are 4 and 6. Achieving coordination results in changes both in the properties of the central atom and the properties of ligand [3,4,5,].

Ortho-phenantroline (1,10 phenantroline) is an organic reagent ($M_r$ = 198,22 g/mol). It is used for photometric determination of the following elements: Cu (II) at wavelength of 272 nm, Cu(I) (435 nm); Co(226 and 270 nm); Cd (226 and 270 nm); Fe(II) (508 nm); Mn (226 and 268 nm); Ru (448 nm); Ni (228 and 270 nm) and Zn (226 and 270 nm) [4,6,7,8,9, 10,11,12].

Together with orto-phenantroline, Fe (II) forms a stable complex of red colour, which is called Ferrand. Color of the complex is stable in the interval of pH = 2-9 [10,11,12].

Smaller cations and bivalent metals obstruct the process of determining iron using ortho-phenantroline complex, forming complexes or hardly soluble compounds Fe(II), Co(II), Ni(II), Sn(II) and Cu(II).

Since ferrous iron is determined by the method, therefore ferric iron reduced to ferrous using hydroxylamine hydrochloride solution [4,5,6,8,9, 10,11,12].

Determination of iron with 1,10–phenanthroline depends on the composition of the composites. The composites used in this paper are alumina and zeolite. Alumina or aluminum oxide, which is used in this paper, is used for aluminum production. It contains 98.6% min $Al_2O_3$, and the rest is impurities Fe, Si, V, F, P, Cr, Na, Zn, etc. Synthetic zeolite is a crystalline sodium aluminosilicate with three-dimensional structure type LTA (zeolite type 4A). The primary building units are atoms of silicon and aluminum, located in the center of the tetrahedron on whose corner are oxygen atoms. Tetrahedra of silica $[SiO_4]^{4-}$ and aluminum $[AlO_4]^{5-}$ are connected by oxygen atoms, so that the tetrahedra are connected to each other by oxygen bridge. Tetrahedra as the primary structure building units can be connected in 4- or 6- rings. By combining tetrahedron in other structural forms formed the so-called secondary building units, which participate in the construction of various types of zeolites. Impurities present in the alumina are also in the zeolite [13].

**Experimental part**

For the experimental part of the work powders of alumina and synthetic zeolite manufactured in The Alumina Factory "Birač" AD, were used. Alumina solution was prepared by mixing 2, 3 and 4 g of alumina with flux ($H_3BO_3$ and $Na_2CO_3$). After that the sample was melted at a temperature of $400^0C$, then again at $1000^0C$. The melted sample was cooled down and dissolved in destilled water, in which $H_2SO_4$ (1:1) was added, after which the solution was transferred into the volumetric flask of 200 $cm^3$. This solution is the basic alumina solution for the determination of iron.

Zeolite solution was prepared by dissolving (1.0, 1.5, 2.0, 3.0) g of zeolite in HCl, after which the solution is transferred into the volumetric flask of 250 $cm^3$. This solution is the basic solution of zeolite for the determination of iron in zeolite.

For the determination of iron in alumina and zeolite spectrometric method was used R805-ISO, which is identical to the methods GOST 13583.3-70, JUS H.B8.057, JUS H.B8.059, and based on the previous reduction of ferric iron using hydroxylamine hydrochloride, and then on the formation of the red tris-1, 10 – phenanthroline iron (II) complex under strictly defined pH (3,5-4,2) and photometric determination at a wavelength of 511 nm[10,11,12].

UV/VIS spectrophotometer Perkin Elmer (Lambda 25) was used for the spectrophotometric determination.

Selection of wavelength of 511nm was performed as follows: Absorption spectra of prepared standard solution of iron were recorded in the range of 300-600 nm. After that was determined absorption maximum, so for the wavelength of this maximum in accordance with Berr's law presents a linear dependence of absorbance on concentration and the steeper linear relation was selected.

Used chemicals pro analysis (p.a.) are: hydroxylamine hydrochloride (Lachner-Czech Republic), 1% ortho-phenanthroline solution (Merck-Germany), 0,25% buffer pH 5,5 (200g $CH_3COONa \cdot 3H_2O$ or 120,56 g $CH_3COONa$ in 500cm$^3$ of water was dissolved with the addition of 12,5 cm$^3$ of concentrated $CH_3COOH$, all transferred to the volumetric flask 1000 cm$^3$ and fill with distilled water), a producer of $CH_3COONa$ Lachner-Czech Republic, and $CH_3COOH$ Zorka-Serbia; a standard solution of iron 0,01 g $Fe_2O_3$ (0,2000 g $Fe_2O_3$ dissolved in 20 cm$^3$ of HCl 1:1 and transferred to a volumetric flask 1000 cm$^3$, then 25cm$^3$ of the solution is transferred to the volumetric flask of 500cm$^3$), producer Merck-Germany and HCl Zorka Serbia-, sulfuric acid 1:1 (Lachner-Czech), $H_3BO_3$ (Zorka-Serbia), $Na_2CO_3$ (Lachner-Czech).

**Results and Discussion**

Stability of the tris-1, 10 – phenanthroline iron (II) complex was monitored in solutions containing different concentrations of alumina (2, 3 and 4) g/200cm$^3$ in the time interval 0-60 minutes. A direct proportionality between the concentration of alumina and iron concentrations was observed, which actually means that the increase of the concentration of alumina in the solution increases the concentration of iron. For the alumina solution containing 2g/200 cm$^3$ of alumina, tris-1, 10 – phenanthroline iron (II) complex is completely stable for the first ten minutes, in the intervals of 10-20 minutes absorbance changes, reaching the initial value once more at the end (Table 1).

For the solution containing 3 and 4g/200cm$^3$ of alumina, tris-1, 10 – phenanthroline iron (II) complex is considerably stable depending on time, and absorbance does not change. There are certain negligible deviations here that can be seen as permissible errors in measurements.

Table 1.

With the concentration of alumina 2g/200cm$^3$ deviations were higher, suggesting that greater errors occur in during determination of very low concentrations of $Fe_2O_3$ (Table 1, Figure 1). Based on the obtained values of relative error of determining it is observed that the relative error of determining is inversely proportional to the concentrations of alumina solution, i.e. the increase of the concentration of alumina in the solution results in relative error decrease. Based on these absorbance measurements in alumina solution, it has been shown that the tris-1, 10 – phenanthroline iron (II) complex is very stable; therefore, the method of determining the stability of the mentioned complex of iron with 1,10–phenanthroline can line up as the method of choice (Figure 1).

Figure 1.

Figure 2.

The values of relative error measurements after 20 minutes for different concentrations of alumina are the following:

- 2g/200cm$^3$ - 2%,
- 3g/200cm$^3$- 1,2%, and
- 4g/200cm$^3$ – 0,7%.

For the solutions of zeolite with concentrations 1,0 and 2,0 g/250 cm$^3$ it was observed that the absorbance in the initial period is changing in a negligibly small degree. After 12 minutes following the addition of ortho-phenantroline, absorbance begins to rise gradually, reaching a maximum at 30 min., after which it decreases again. In the solution containing 1,5 g/250 cm$^3$ of zeolite, no considerable increase in absorbance can be observed up to 12 minutes from the moment when ortho-phenantroline was added; however, it rises continuously thereafter, which indicates that probably another complex is formed and disturb the formation of the tris-1, 10 – phenanthroline iron (II) complex, because in zeolite solution is present more ions compared to alumina, which disturb complex formation**.** (Table 2). However, the solution containing 3g/250cm3 of zeolite the complex is stable for 10 minutes, after which the absorbance begins to rise, though not significantly, indicating that there was not enough ortho-phenantroline to develop the color and disturb complex formation with ions from solution is greater, because the content of zeolite in the solution was too high (Table 2 , Fifure 2). To determine the calibration curve for iron, 0.2 g/dm$^3$ $Fe_2O_3$ solution was used, from which solutions 1, 2, 3, 4, 5 and 6 solutions were made.

Just as with determining the concentration of the sample, in this case the absorbance of each standard solution of different concentrations (0,02; 0,04; 0,05; 0.06; 0,08 and 0,10) mg $Fe_2O_3$/cm$^3$ was measured at the same time interval (0-60 min.) (Table 3).

Based on the obtained data, stability of the solution in the examined period is observed. The increase of the concentration of the standard $Fe_2O_3$ solution results in the increase of the absorbance and concentration of $Fe_2O_3$, which means that there is a direct dependence between the concentration of the standard solution and absorbance. The value of absorbance and the concentration of the standard solution are constant over time, which justifies the use of spectrophotometric methods.

Table 2.

Using the obtained values of the absorbance of the standard solution, measured for 10 minutes from the moment of adding ortho-phenantroline for each solution, and applying the formula K = C/A, calibration constant is obtained. Based on the obtained values of constants, the average value of the constant is calculated at 0,1412.

Based on all the results of zeolite absorbance, it can be concluded, that the tris-1, 10 – phenanthroline iron (II) complex is stable in a narrow time interval, after which the increase in absorbance occurs, reaching a maximum after 30 minutes from the moment ortho-phenantroline had been added, which indicates that most probably occurred disturb of the tris-1, 10 – phenanthroline iron (II) complex formation and probably formation of new complex with ortho-phenantroline

Table 3.

This complex disintegrates after a certain period of time, which is indicated by the decrease of absorbance after 30 minutes.

Oscillations of the absorbance depending on the time for those concentrations of zeolite suggest that in this composite the formation of the complex is disturb, probably caused by the composite structure and present anions of mineral acids used in their dissolution. This could be the subject of new research in the field of stereochemistry. Complexing of some other substance in the zeolite must have taken place, since it is a complex compound and the interactions of ortho-phenantroline complex with other components in the solution of these composites are possible.

In the case of the solution prepared from alumina, absorbance were more stable, due to less frequent interactions between the tris-1, 10 – phenanthroline iron (II) complex and other components present in the composite. During the spectrophotometric determination of iron under strictly defined pH values, wavelength and concentration regime, it was determined that determinations are limited, partly due to the stability of the complex, and partly due to disturb complex formation with components that are present in the composite. In determining the concentration of the standard solution of iron with ortho-phenantroline, absorbance was stable, because there were no impurities that could influence on the formation and stability of tris-1, 10 – phenanthroline iron (II) complex, which goes in favor with the fact that with complex solutions, due to the fact that absorbance changes, a new complex is formed, with presence of composite components, and some of them disturb of tris-1, 10 – phenanthroline iron (II) complex formation.

Due to all these facts, spectroscopic methods of determining iron in the zeolite cannot be the method of choice, since tris-1, 10 – phenanthroline iron (II) complex is stable in a very short time interval.

**Conclusions**

1. Tris-1, 10 – phenanthroline iron (II) complex obeys the Beer's law (linear function of the dependence between absorbance and concentration) for all tested solutions.
2. For the concentration of alumina 2g/200 $cm^3$ , tris-1, 10 – phenanthroline iron (II) complex of iron is unstable in the time interval of 10-20 minutes, which explains the low concentration of iron, which results in large error in determining.
3. At the concentration of alumina (3, 4) g/200$cm^3$, tris-1, 10 – phenanthroline iron (II) complex of iron is stable throughout the entire time interval, and the error in determining is reduced, which indicates that the concentration of alumina is suitable for the determination of iron using this method.
4. For different concentrations of zeolite (1; 1,5; 2.0 and 3.0) g/250$cm^3$ in the time interval of 0-60 minutes from the moment of adding ortho-phenantroline, stability of the tris-1, 10 – phenanthroline iron (II) complex occurs in a short time interval (8-10 minutes), as absorbency further increases, which gives unreliable results of determination, so this method of determining iron in zeolite cannot be the method of choice because there is disturb of complex formation and probably the formation of a new complex with components of the composite.
5. During spectrophotometric determination of the standard solution of iron with ortho-phenantroline, absorbance does not change for the whole period, i.e., the

complex is stable, which goes in favour with the idea that the composition of the composite influences the stability of the complex.

6. Spectrophotometric determinations of iron with ortho-phenantroline under strictly defined pH values, wavelength and concentration regime, are limited to the composition of composites (zeolite and alumina) and present mineral acid anions used for dissolution there of.
7. Conclusions five and six indicate that the future research should focus on research stereochemistry resulting complex from these composite zeolites and alumina.

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Table 1. Dependence between absorbance and concentration of $Fe_2O_3$ for different alumina concentrations (2, 3 i 4 g/200 $cm^3$) and time

| time development colour (min) | Content $Fe_2O_3$ solution 1($R_1$) | | relative error conc. % | Content $Fe_2O_3$ solution 2($R_2$) | | relative error conc. % | Content $Fe_2O_3$ solution 3($R_3$) | | relative error conc. % |
|---|---|---|---|---|---|---|---|---|---|
| | A | C (mg/$cm^3$) | | A | C (mg/$cm^3$) | | A | C (mg/$cm^3$) | |
| 2 | 0,240 | 126,51 | 2,9 | 0,415 | 150,80 | 2,8 | 0,729 | 201,35 | 0,20 |
| 4 | 0,240 | 126,51 | 2,9 | 0,408 | 148,16 | 1,1 | 0,731 | 201,92 | 0,10 |
| 6 | 0,240 | 126,51 | 2,9 | 0,405 | 147,03 | 0,3 | 0,729 | 201,35 | 0,20 |
| 8 | 0,240 | 126,51 | 2,9 | 0,405 | 147,03 | 0,3 | 0,736 | 203,33 | 0,80 |
| 10 | 0,242 | 127,64 | 2,0 | 0,406 | 147,41 | 0,6 | 0,737 | 203,61 | 0,90 |
| 12 | 0,269 | 142,89 | 9,5 | 0,405 | 147,03 | 0,3 | 0,728 | 201,07 | 0,30 |
| 14 | 0,263 | 139,51 | 7,1 | 0,403 | 146,28 | 0,2 | 0,728 | 201,07 | 0,30 |
| 16 | 0,256 | 135,55 | 4,0 | 0,403 | 146,28 | 0,2 | 0,733 | 202,48 | 0,40 |
| 18 | 0,253 | 133,86 | 2,7 | 0,404 | 145,52 | 0,7 | 0,730 | 201,63 | 0,00 |
| 20 | 0,249 | 127,64 | 2,0 | 0,404 | 144,77 | 1,2 | 0,739 | 203,05 | 0,70 |
| 25 | 0,247 | 126,51 | 2,9 | 0,403 | 146,39 | 1,5 | 0,729 | 200,22 | 0,70 |

*Where: solution1($R_1$)- concentration of alumina* (2g/200 $cm^3$), *solution2($R_2$)- concentration of alumina*(3g/200 $cm^3$), *solution3($R_3$)- concentration of alumina* (4g/200 $cm^3$).

Table 2. Dependence between absorbance and concentration of $Fe_2O_3$ for different zeolites concentrations (1,0; 1,5; 2,0 and 3,0 g/250 $cm^3$) and time.

| time development colour min. | Content $Fe_2O_3$ solution 1($R_1$) | | relative error conc. % | Content $Fe_2O_3$ solution 2($R_2$) | | relative error conc. % | Content $Fe_2O_3$ solution 3($R_3$) | | relative error conc. % | Content $Fe_2O_3$ solution 4($R_4$) | | relative error conc. % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | A | C mg/$cm^3$ | | A | C mg/$cm^3$ | | A | C mg/$cm^3$ | | A | C mg/$cm^3$ | |
| 2 | 0,195 | 139,57 | 9,2 | 0,269 | 133,57 | 13,7 | 0,362 | 136,08 | 30,8 | 0,472 | 119,15 | 4,45 |
| 4 | 0,196 | 140,35 | 8,7 | 0,272 | 135,12 | 12,7 | 0,369 | 138,80 | 29,4 | 0,476 | 120,19 | 3,6 |
| 6 | 0,201 | 144,22 | 6,2 | 0,273 | 135,63 | 12,4 | 0,375 | 141,12 | 28,2 | 0,480 | 121,22 | 2,8 |
| 8 | 0,202 | 145,00 | 5,7 | 0,277 | 137,71 | 11,0 | 0,387 | 145,77 | 25,9 | 0,485 | 122,51 | 1,8 |
| 10 | 0,204 | 146,55 | 4,7 | 0,280 | 139,26 | 10,0 | 0,433 | 163,61 | 16,8 | 0,488 | 123,29 | 1,1 |
| 12 | 0,206 | 148,10 | 3,6 | 0,284 | 141,33 | 8,7 | 0,533 | 202,46 | 3,0 | 0,492 | 124,32 | 0,3 |
| 14 | 0,210 | 151,20 | 1,6 | 0,293 | 145,99 | 5,7 | 0,559 | 212,46 | 8,1 | 0,493 | 124,58 | 0,1 |
| 16 | 0,219 | 158,18 | 2,9 | 0,297 | 148,06 | 4,3 | 0,606 | 230,68 | 17,3 | 0,497 | 125,61 | 0,7 |
| 18 | 0,223 | 161,28 | 4,9 | 0,301 | 150,14 | 3,0 | 0,632 | 240,76 | 22,5 | 0,499 | 126,13 | 1,2 |
| 20 | 0,229 | 165,93 | 8,0 | 0,307 | 153,24 | 1,0 | 0,659 | 251,23 | 27,8 | 0,503 | 127,17 | 2,0 |
| 25 | 0,233 | 169,04 | 10,0 | 0,328 | 164,11 | 6,0 | 0,616 | 234,56 | 19,3 | 0,505 | 127,68 | 2,4 |
| 30 | 0,247 | 179,89 | 17,0 | 0,350 | 175,50 | 13,4 | 0,582 | 221,38 | 12,6 | 0,510 | 128,97 | 3,4 |
| 60 | 0,207 | 148,88 | 3,1 | 0,499 | 252,64 | 63,2 | 0,488 | 184,93 | 5,9 | 0,515 | 130,27 | 4,5 |

*Where: solution1($R_1$)- concentration of zeolite*(1,0g/250 $cm^3$), *solution1($R_2$)- concentration of zeolite*(1,5g/250 $cm^3$), *solution3($R_3$)- concentration of zeolite*(2,0g/250 $cm^3$), *solution4($R_4$)- concentration of zeolite*(3,0g/250 $cm^3$).

Table 3. Dependence between absorbance and concentration of $Fe_2O_3$ and time for a standard solution of 0,2 g/$dm^3$ $Fe_2O_3$

| time development colour min. | $Fe_2O_3$ content, mg/$cm^3$ | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | solution 1 | | solution 2 | | solution 3 | | solution 4 | | solution 5 | | solution 6 | |
| | A | C mg/$cm^3$ | A | C mg/$cm^3$ | A | C mg/$cm^3$ | A | C mg/$cm^3$ | A | C mg/$cm^3$ | A | C mg/$cm^3$ |
| 2 | 0,142 | 0,020 | 0,283 | 0,040 | 0,354 | 0,050 | 0,425 | 0,060 | 0,566 | 0,080 | 0,708 | 0,100 |
| 4 | 0,142 | 0,020 | 0,284 | 0,040 | 0,355 | 0,050 | 0,426 | 0,060 | 0,568 | 0,080 | 0,710 | 0,100 |
| 6 | 0,143 | 0,020 | 0,286 | 0,040 | 0,357 | 0,050 | 0,428 | 0,060 | 0,571 | 0,081 | 0,714 | 0,100 |
| 8 | 0,144 | 0,020 | 0,286 | 0,040 | 0,357 | 0,050 | 0,428 | 0,060 | 0,571 | 0,081 | 0,714 | 0,100 |
| 10 | 0,142 | 0,020 | 0,283 | 0,040 | 0,354 | 0,050 | 0,425 | 0,060 | 0,566 | 0,080 | 0,708 | 0,100 |
| 12 | 0,142 | 0,020 | 0,284 | 0,040 | 0,355 | 0,050 | 0,426 | 0,060 | 0,568 | 0,080 | 0,710 | 0,100 |
| 14 | 0,141 | 0,020 | 0,282 | 0,040 | 0,353 | 0,050 | 0,424 | 0,060 | 0,565 | 0,080 | 0,706 | 0,100 |
| 16 | 0,142 | 0,020 | 0,283 | 0,040 | 0,354 | 0,050 | 0,425 | 0,060 | 0,566 | 0,080 | 0,708 | 0,100 |
| 18 | 0,141 | 0,020 | 0,282 | 0,040 | 0,352 | 0,050 | 0,422 | 0,060 | 0,563 | 0,079 | 0,704 | 0,100 |
| 20 | 0,142 | 0,020 | 0,283 | 0,040 | 0,354 | 0,050 | 0,425 | 0,060 | 0,566 | 0,080 | 0,708 | 0,100 |
| 25 | 0,142 | 0,020 | 0,283 | 0,040 | 0,354 | 0,050 | 0,425 | 0,060 | 0,566 | 0,080 | 0,708 | 0,100 |
| 30 | 0,141 | 0,020 | 0,282 | 0,040 | 0,352 | 0,050 | 0,422 | 0,060 | 0,563 | 0,079 | 0,704 | 0,100 |
| 60 | 0,141 | 0,020 | 0,282 | 0,040 | 0,352 | 0,050 | 0,422 | 0,060 | 0,563 | 0,079 | 0,704 | 0,100 |

*Where: solution 1 - 0,02 mg $Fe_2O_3$ /$cm^3$; solution 2 - 0,04 mg $Fe_2O_3$ /$cm^3$; solution 3 - 0,05 mg $Fe_2O_3$ /$cm^3$; solution 4 - 0,06 mg $Fe_2O_3$ /$cm^3$; solution 5 - 0,08 mg $Fe_2O_3$ /$cm^3$; solution 6 - 0,10 mg $Fe_2O_3$ /$cm^3$.*

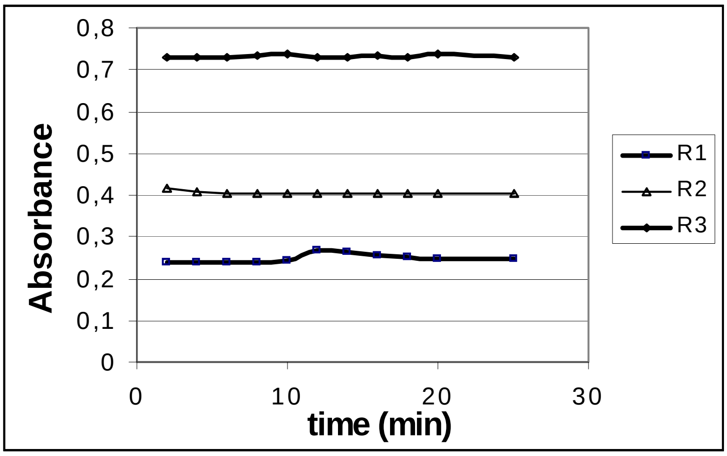


Figure 1.

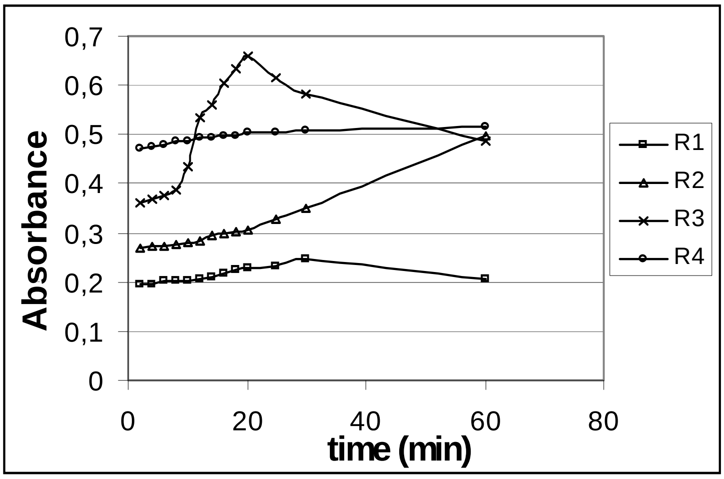


Figure 2.

Figure 1. Dependence of absorbance of the time for different concentrations alumina (2, 3 and 4)g/200cm$^3$, solutions $R_1$, $R_2$, $R_3$.

Figure 2. Dependence of absorbance of the time for different concentrations zeolite (1,0; 1,5; 2,0 and 3,0)g/250cm$^3$, solutions $R_1$, $R_2$, $R_3$ and $R_4$.

Table 1. Dependence between absorbance and concentration of $Fe_2O_3$ for different alumina concentrations (2, 3 and 4 g/200 cm$^3$) and time.

Table 2. Dependence between absorbance and concentration of $Fe_2O_3$ for different zeolites concentrations (1, 1.5, 2.0 and 3.0 g/250 cm$^3$) and time.

Table 3. Dependence between absorbance and concentration of $Fe_2O_3$ and time for a standard solution of 0,2 g/dm$^3$ $Fe_2O_3$.