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## Introduction

The pollution of water and soil from industrial sources by certain chemical products (hydrocarbons, metals ...) or agriculture (pesticides, fertilizer, ...) is a source of environmental degradation. Recent decades have seen the emergence of new application areas of transition metal complexes such use in soil remediation.

The objective of this work is to test the ability of certain metal coordination compounds in the decontamination of sludge produced by NAFTAL : the principal company selling petroleum-based fuels for domestic consumption in Algeria. The studied sludge consists of water, hydrocarbons, oils, solvents and heavy metals with a high percentage of lead.

A series of new manganese(II), iron(II), copper(II) and zinc(II) complexes with glucose product of starch hydrolysis (in a basic medium and in the presence of metal ion) contained in a natural product of wide consumption noted X. They have been prepared and characterized by elemental analysis, emission atomic spectroscopy, TGA measurements, FTIR, UV-Vis spectra and conductivity. The complexes are respectively noted C1, C2, C3 and C4.

## Structural Characterization

The complexing solution is obtained by infusion of product X in hot water and cooled to room temperature. This solution is then added to a metal solution whose pH is kept at 11 by adding a few drops of ammonia.

Figure 1 represents FTIR superimposed spectra of the evaporated complexing solution and starch [1]. Examination of the figure shows that the two spectra describe the same organic entity.

## Complexing compound

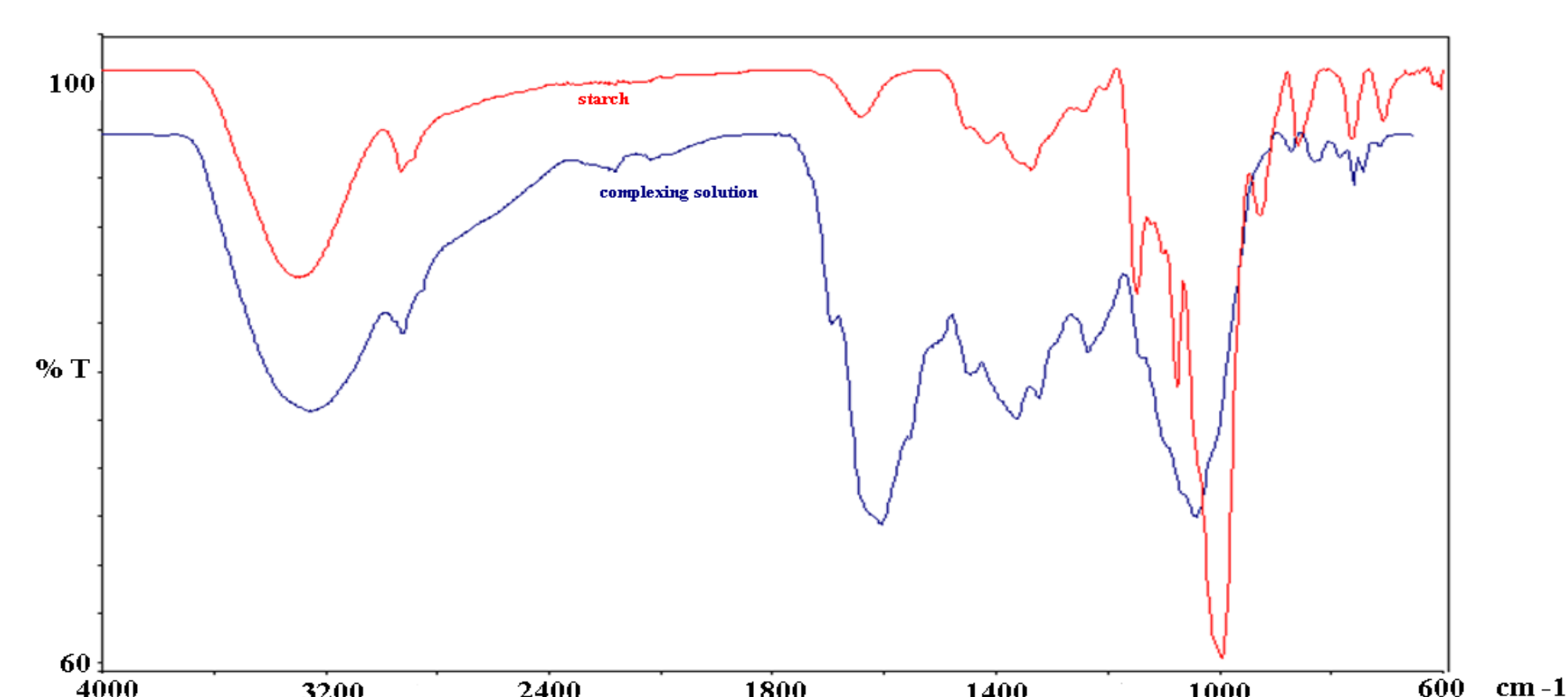


Fig1. FTIR Spectra.

The FTIR spectra of the complexes (Fig. 2) when are compared to glucose one confirm that the complexing molecule is effectively glucose that we note HL<sup>1</sup>.

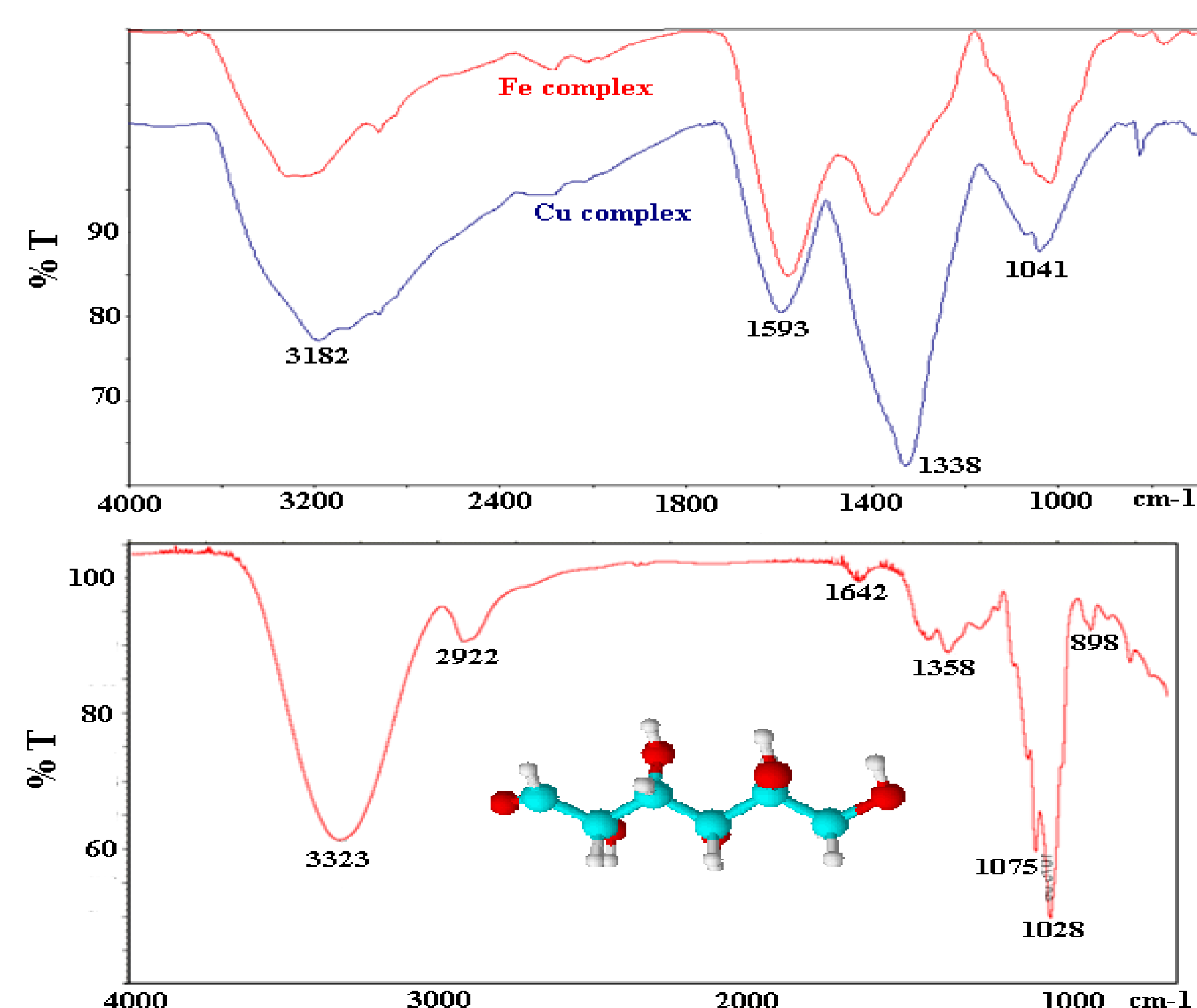


Fig.2. FTIR spectra of Fe and Cu complexes and glucose [2].

## Complexes

The physical properties and analytical data of the complexes are summarized in Table below. The synthesized complexes are totally soluble in DMSO, DMF and acetonitrile, partially soluble in water, ethanol and methanol. Their molar conductance data in DMSO solution revealed that they are non electrolyte species. Mass spectra of all the compounds stand in good agreement with proposed structure.

	Formula	Molecular mass	Melting point (°C)	$\Lambda_{DMF}$ (ohm <sup>-1</sup> .cm <sup>2</sup> .mol <sup>-1</sup> )
C1	Mn(L <sup>1</sup> ) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub>	450	>400	31.67
C2	Fe(L <sup>1</sup> ) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub>	450	183	37.80
C3	Cu(L <sup>1</sup> ) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub>	458	143	29.13
C4	Zn(L <sup>1</sup> ) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub>	460	>400	31.69

## By IR

The infrared spectra assignment of the proposed complexes structures was made through consideration of their infrared spectra. The IR spectra provide valuable information regarding the nature of the functional group attached to the metal atom. The coordinated stretching vibration bands of the isolated products were assigned comparing the IR spectra of the free ligand to the spectra of its metal complexes.

## By UV-visible

Examination of the UV-visible spectra of the complexes supports completely the proposed structural hypotheses, namely:

- An octahedral environment for the Mn (II) in complex C1
- An octahedral environment for Fe (II) in complex C2
- A square planar environment for Cu (II) in complex C3
- A tetrahedral environment for Zn (II) in the complex C4

## Thermal Analysis

The results of TGA analysis when are compared to that of glucose [3](Fig. 3b) allowed to determine the nature of water molecules in complex and the different steps of decomposition within complexes. The thermogram of C3, given as example (Fig. 3a) shows a first decomposition in the region 50–110° C, which is due to the loss of lattice water molecules. The temperature range of 200–800° C represents the decomposition of the organic part.

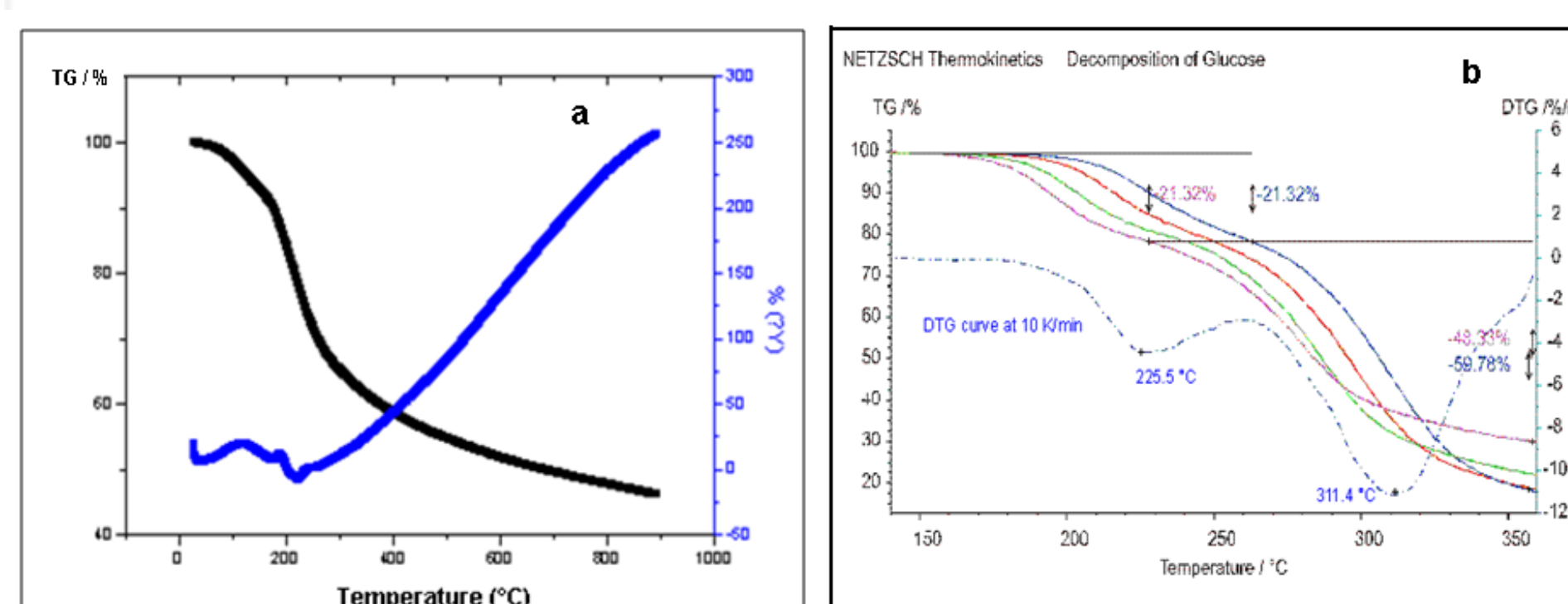


Fig.3: TGA curves.

## Application

The objective of the present study was to investigate the potential use of the studied complexes as a competitive adsorbent material for the removal of lead (II) from aqueous solutions and sludges.

## Synthetic solutions

The results obtained with synthetic solutions are shown in Figure 4 and 5. In case of figure 4, 2 mg of complex is used when 20 mg of adsorbent is used in the case of figure 5. The non removal Pb(II) was determined by Atomic Absorption at 283 nm.

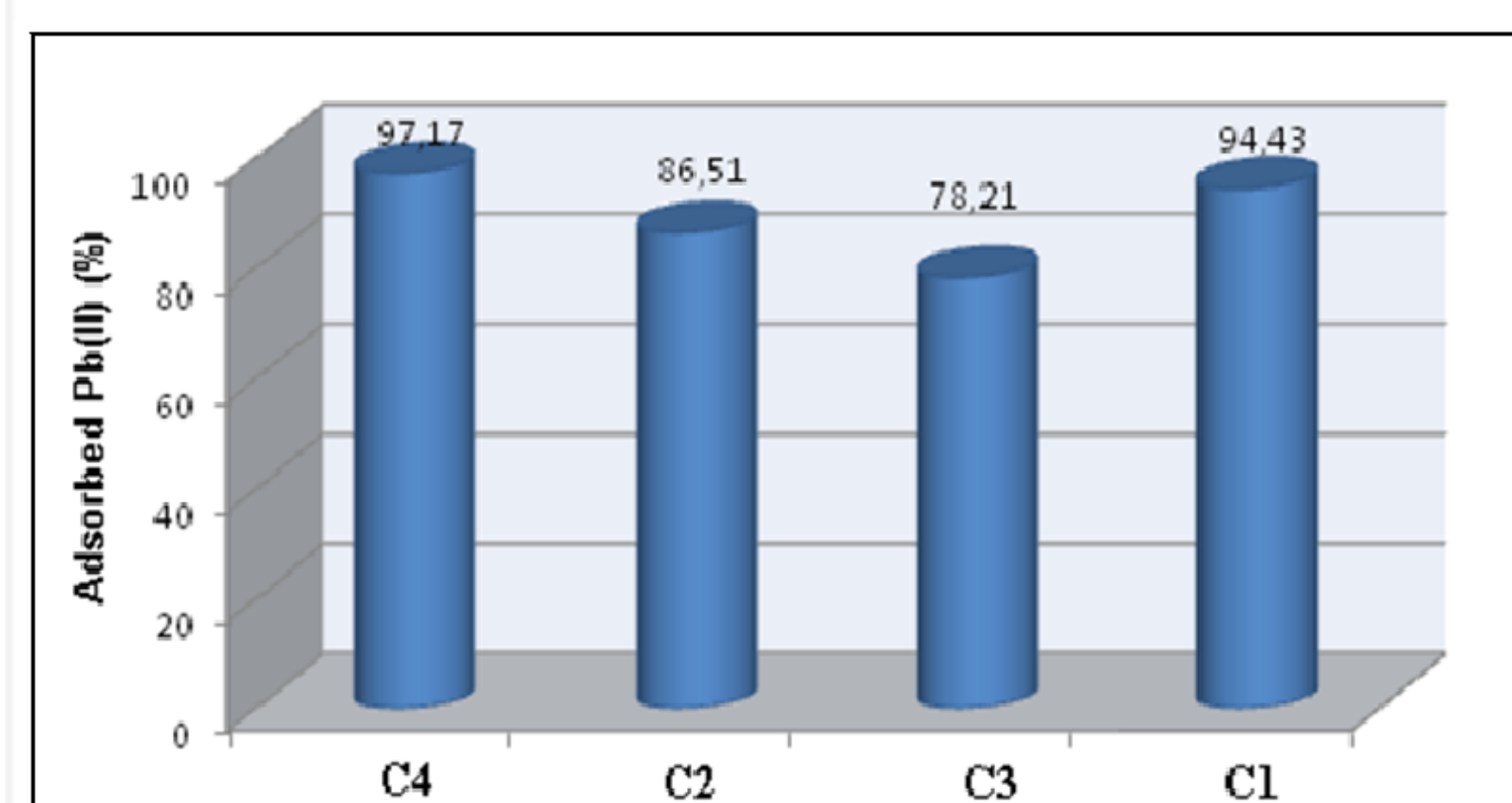


Fig. 4: % adsorbed Pb(II) (2mg of complex).

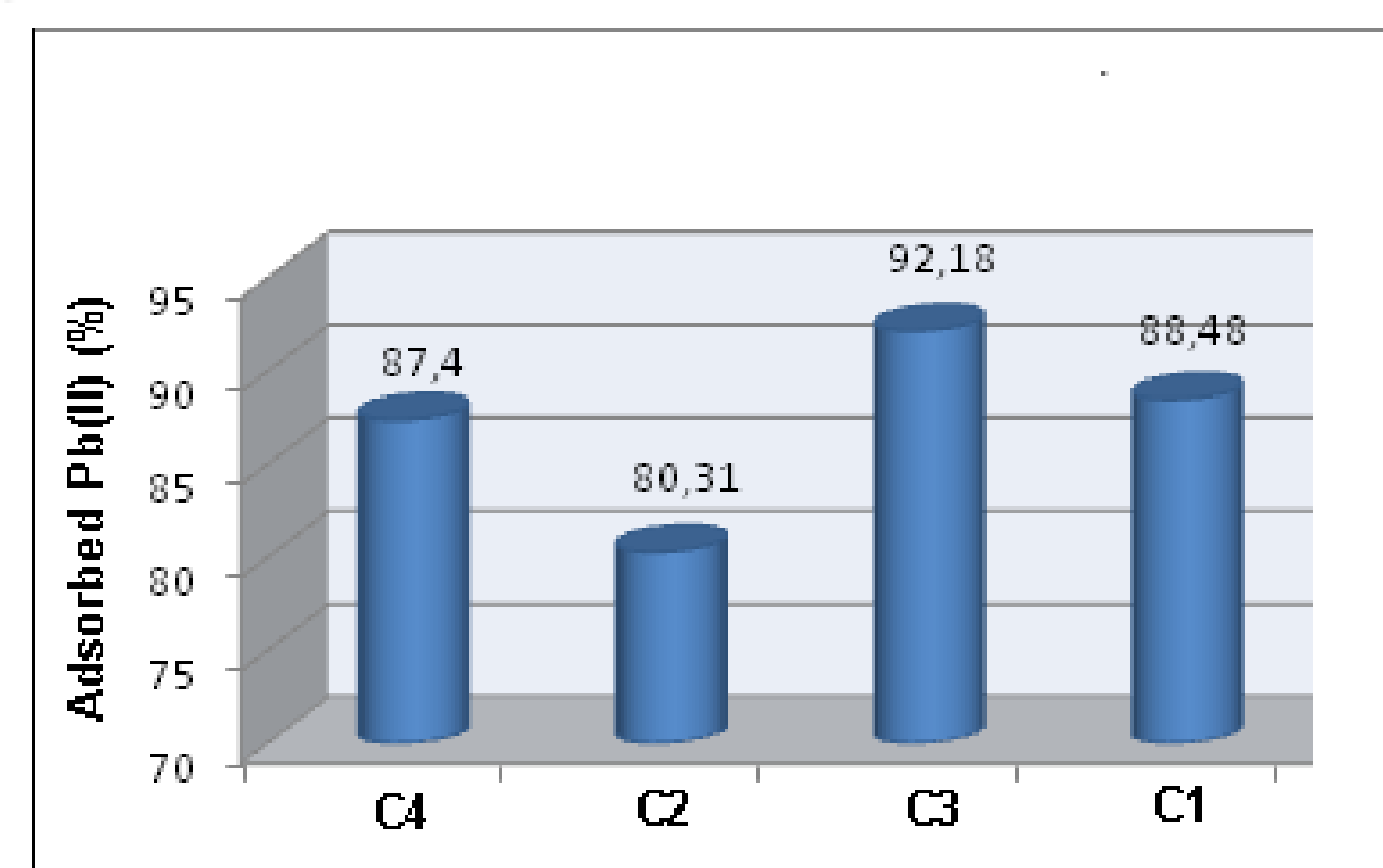


Fig. 5: % adsorbed Pb(II) (20 mg of complex).

Based on these results, it seems that all the complexes showed a very interesting adsorption character.

## Sludge solutions

After the encouraging results obtained with synthetic solutions, we consider appropriate to test our complexes on a waste product containing lead (1 g / kg), ie a hydrocarbon sludge. The objective would be to maximize the absorbency of the synthesized compounds on a real medium. The results are shown in Figure 6.

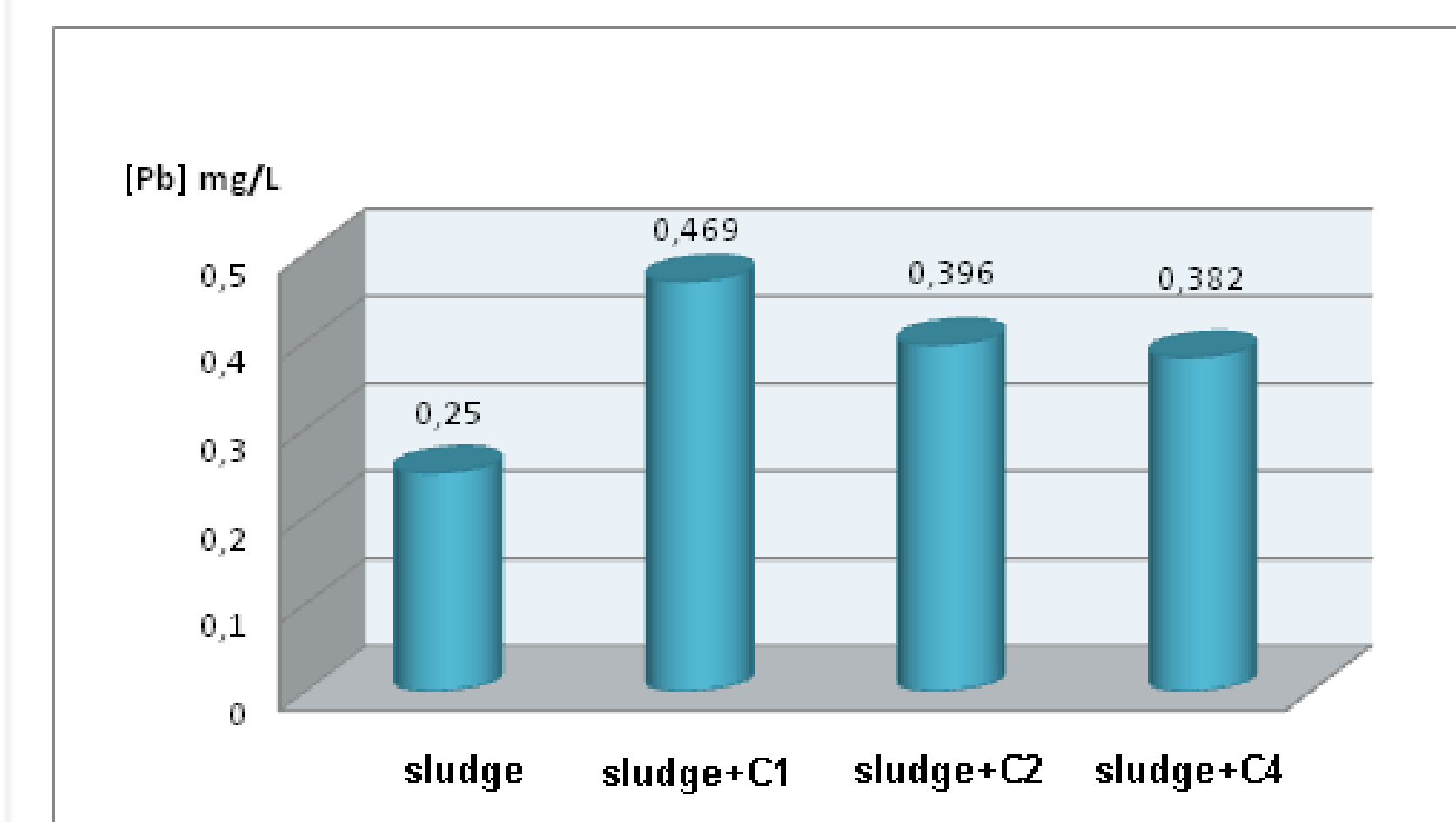


Fig. 6: Sludge solution results.

Examination of these tests showed that in the presence of the sludge, the adsorbent power of complexes is inhibited. Nevertheless, we observed another interesting compartment: the leaching one.

## Conclusions

According to the results obtained in the adsorption process study of Pb (II) on isolated complexes, we can conclude that:

- The compounds used in this study was found to be an effective and low-cost adsorbent for the adsorption of Pb (II) ion only from aqueous solutions.
- Work remains to be done to enhance the leaching power of the complexes in order to provide a decontamination procedure.

## References

- [1] Integrated Spectral Data Base System for Organic Compounds. <http://www.aist.go.jp/RIODB/SDBS/menu-e.html>.
- [2] <http://mail.ypu.edu.tw/~wnhuang/Database/IR/ir.htm>
- [3] [www.netzsch-thermal-analysis.com](http://www.netzsch-thermal-analysis.com)