

**Structural and kinetic characterization of expired drugs** by <sup>1</sup>H NMR relaxometry and FT-IR spectroscopy



# **R.I.** Chelcea<sup>1</sup>, **D.** Danis<sup>1</sup> and **R.** Fechete<sup>1</sup>

<sup>1</sup>Technical University of Cluj-Napoca, Memorandumului Str. No. 28, R-400114, Cluj-Napoca, Romania.

# Introduction

The pollution with expired drug of aquatic environments, in particular rivers and lakes, is becoming a current concern both, nationally and internationally. Thus, one can say that the interest in the exact assessment of the quantity of pharmaceutical waste as well as their management after the expiration date is more and more advanced. For the present study were used: I) expired drugs in the form of: 1) tablets: Paracetamol, Clarinase, SennaLax, Anticârcel, Digest Duo, Tusin, 2) capsules: Diurocard, Omeprazol, DigestDuo and 3) powders: Vitamina C, Ca, Prolyte, as well as II) non expired drugs within the same form of: 1) tablets: Paracetamol, Naproxeno, Azitromicina, Clarinase, SennaLax, Faringonil, Anticârcel, Tusin, 2) capsules: Diurocard, ErceFlora, Omeprazol, DigestDuo and 3) powders: Vitamina C, Ca. FT-IR spectroscopy is used for the structure characterization while the molecular dynamic characterization of expired and in-term drugs was made from <sup>1</sup>H NMR relaxometry data. For that, the recorded NMR signal (CPMG pulse sequence) was analysed by inverse Laplace transform in order to obtain the distribution of the transverse relaxation time, T<sub>2</sub>. The solubility in liquid mediums with different pH was assessed from the kinetic measurements of i) electrical conductivity of a 200 ml solution with adjusted pH and ii) in time repetitive measurement of  $T_2$ -distribution.

### **Expired and in- term drugs used of the study**



### **FT-IR** spectra of expired and in-term drugs



#### **The solubility kinetics measurements by** <sup>1</sup>H NMR **relaxometry**



#### **Electrical conductivity measurements of expired drugs**











1E-3 0.01

 $T_{2}$  [s]

0.1

⊂ 1E-5

°015

1E-4

in-term powders



dissolution time [min.

600

500

ഋ0.10

Ø 0.05

orbar

700

λ [nm]

800

DigestDuo





## Conclusions

1E-3 0.01

 $T_{2}$  [s]

0.1

DigestDu

1E-4

in-term capsules

1E-5

\_\_\_\_15 0

**≥**10

 $\mathbf{X}$ 

For the investigation and characterization of different drugs, advanced methods were used, such as: proton nuclear magnetic resonance (<sup>1</sup>H NMR) combined with Laplace data analysis, FT-IR and VIS-IR-appropriate spectroscopy. Kinetic measurements of electrical conductivity were performed for the assessment of stabilility in aqueous medium with diffrent pH.

1E-5 1E-4

0.05

<u>}</u> 0.04

1E-3 0.01

 $T_{2}$  [s]

0.1

in-term tablets

SenaLa

Changes in the molecular dynamics of drugs were observed after expiration. While those in term presents several immobile components.

The FT-IR spectra for in-term and expired drugs were compared and it was observed that from the structural point of view of the base molecule they are similar.

## **Bibliography**

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