OPTIMIZATION OF AN ON-LINE SPE WITH UPLC-MS/MS METHOD FOR THE DETERMINATION OF ANTINEOPLASTIC COMPOUNDS IN WASTEWATER

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INTRODUCTION

In the last decade the number of works that have detected antineoplastic compounds in samples of environmental waters has increased[1]. Concern over these compounds has grown because the effects they can cause are still unknown, but could be very damaging and even more the mixture of them[2]. Concentrations in wastewater samples are in the range of $ng·L^{-1}$, so it is necessary to use highly sensitive techniques such as UPLC-MS / MS and extraction and preconcentration techniques such as on-line SPE. In this work, all the parameters than affect the process have been optimised (sample volume, load wash solvents, sample loading solvent and pH), for the extraction and determination of Etoposide (ETO), Gemcitabine (GEM), Methotrexate (MET), Tamoxifen (TAM), Cyclophosphamide (CP), Vincristine (VINC) and vinblastine (VINB).

EXPERIMENTAL

Sample loading solvents: Three different loading solvents were tested at 5mL, 4mL, 3mL, 2mL and 1mL of volume of injection:
- Mili-Q water + 0,01% formic acid (v/v) (pH=3,11) (3mL of volume injection)
- Mili-Q water without additives (pH=5,78) (3mL of volume injection)
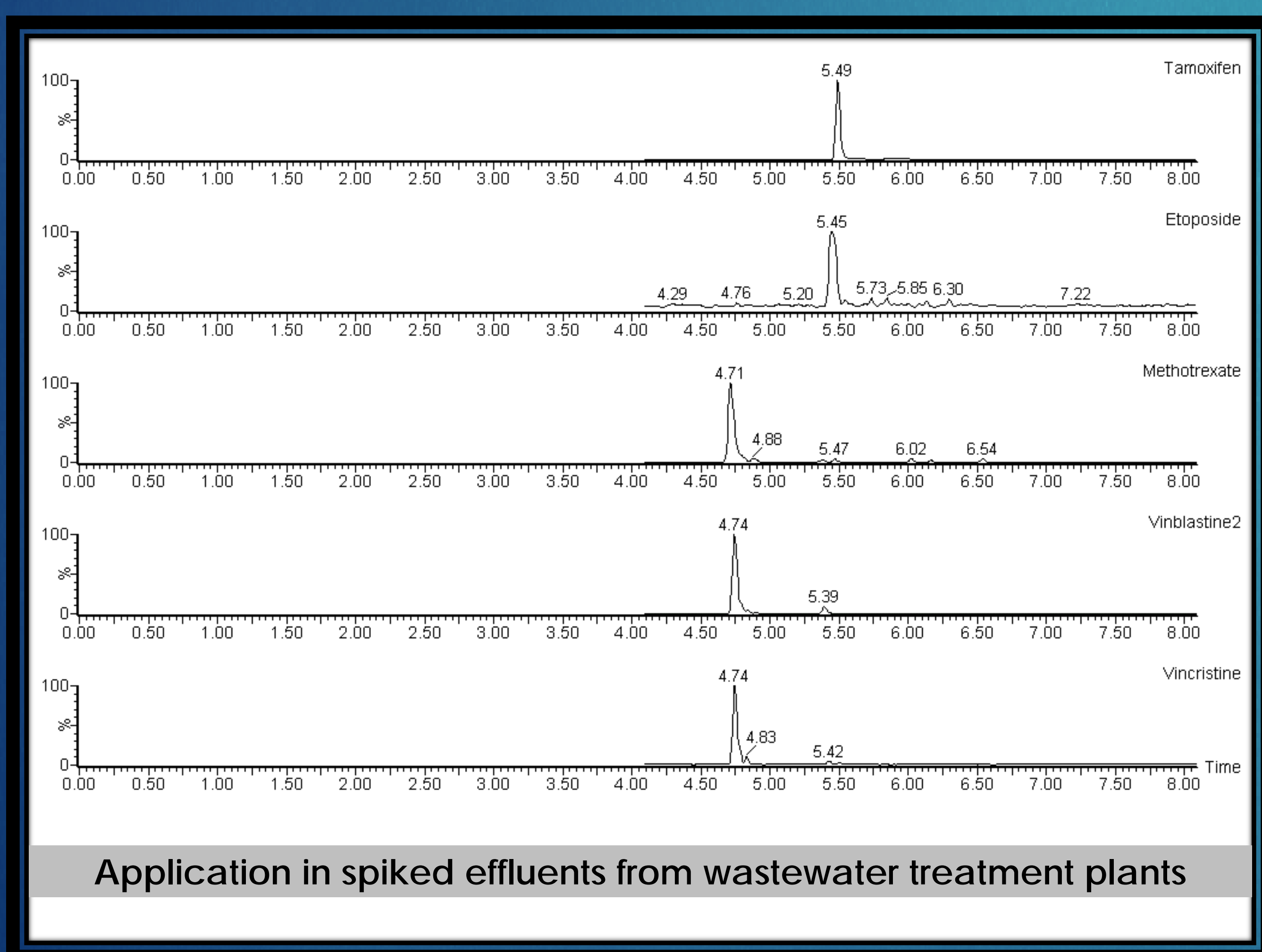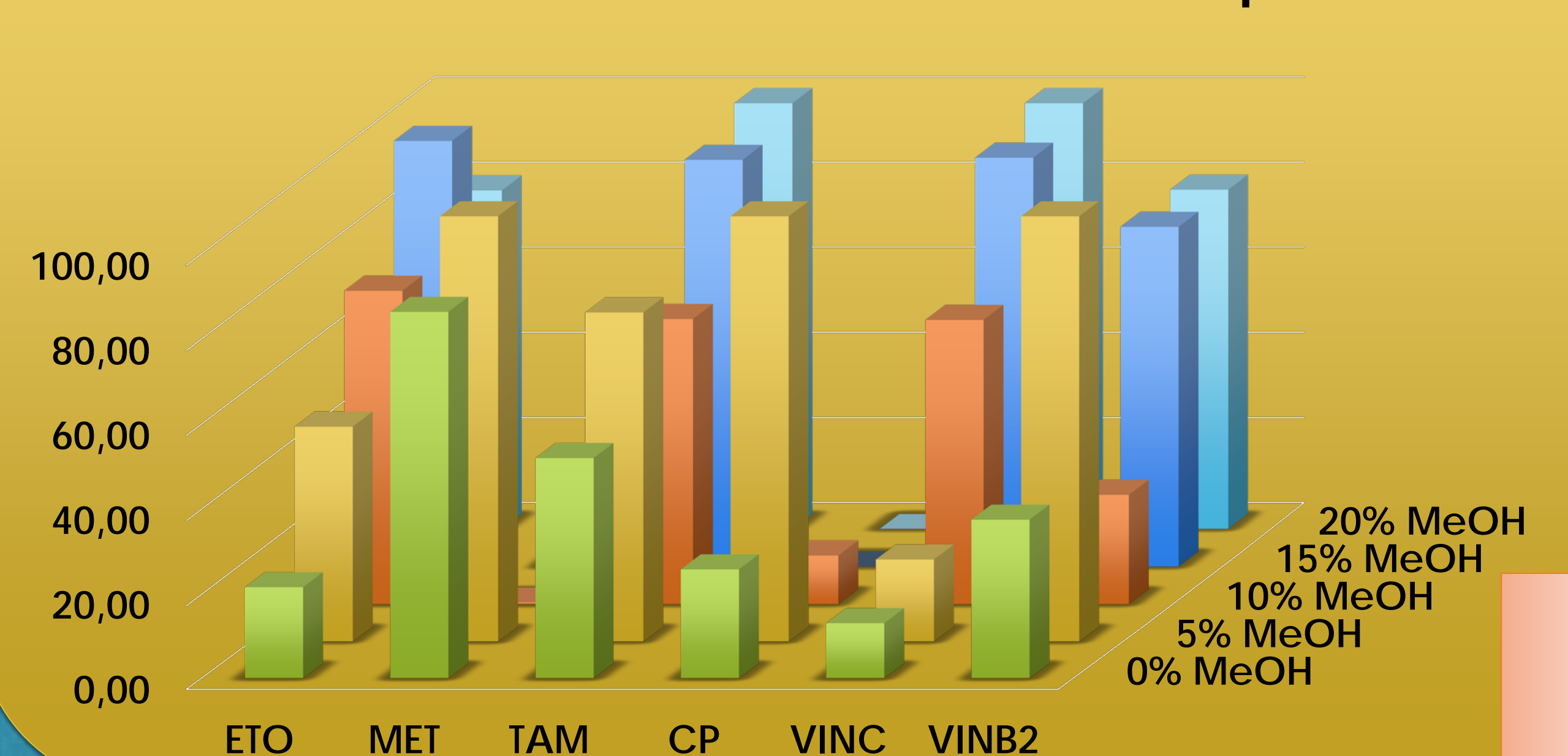- Mili-Q water + 0,1% NH_3 (v/v) (pH=10,67) (5mL of volume injection)

After obtaining the best volume of injection for each phase of load they were compared to know what would be optimal
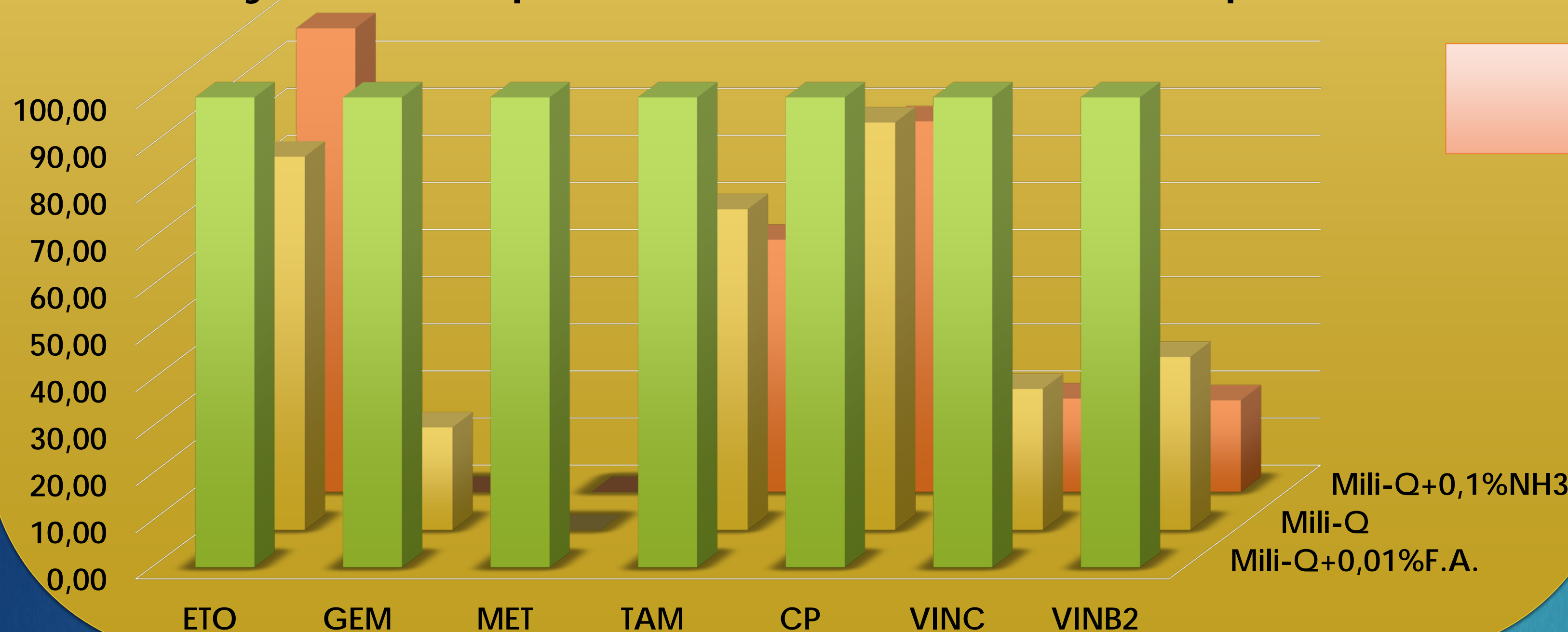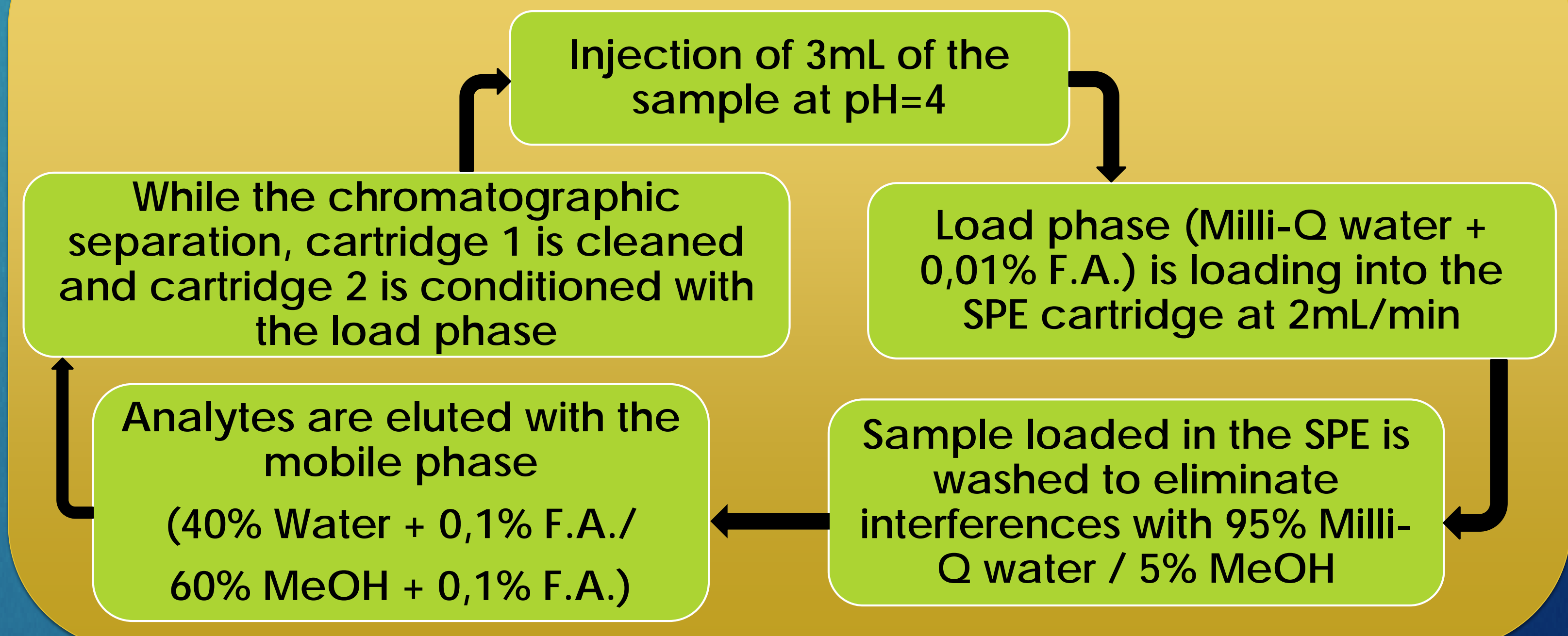

pH of the sample: Three different pH of the sample were tested (4, 7 and 10)
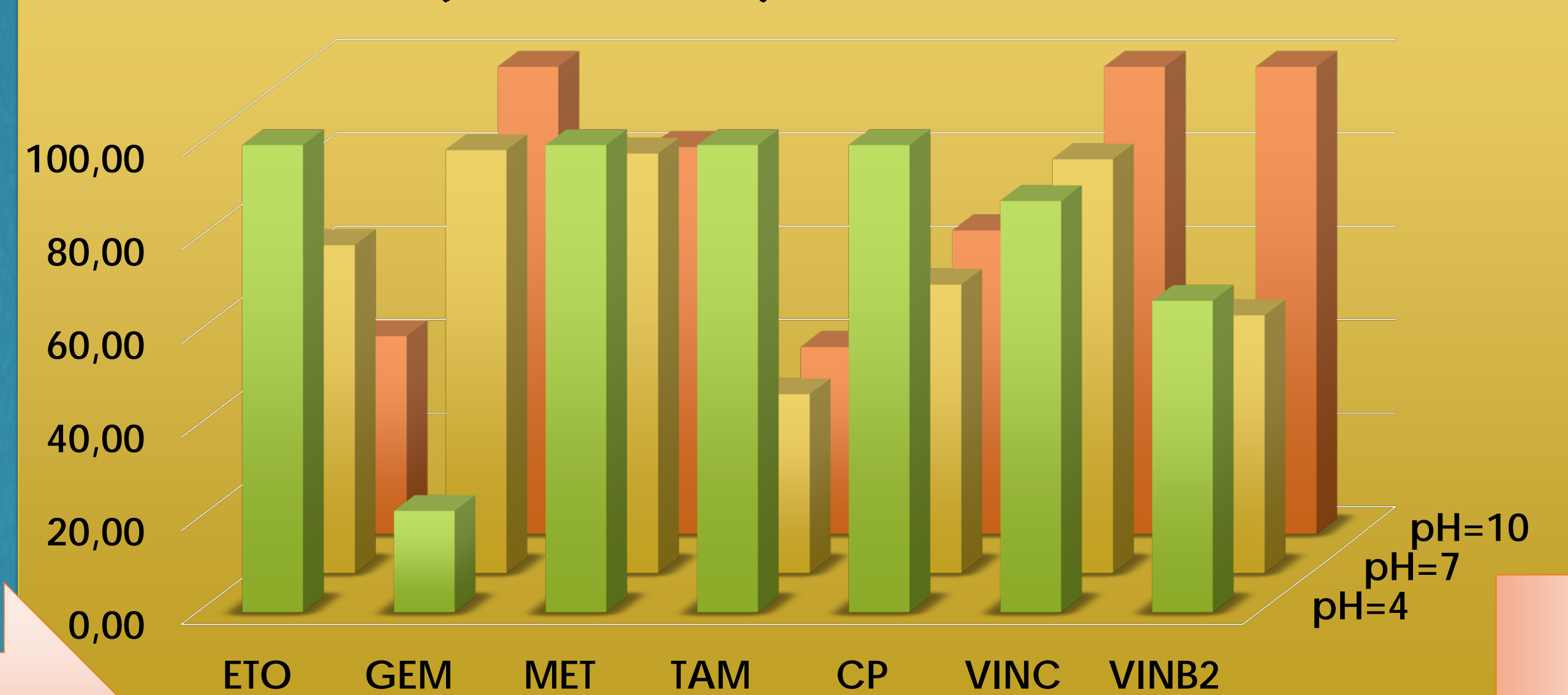
Sample wash step: Different proportions of organic solvent were tested to select the best composition.

Application in spiked effluents from wastewater treatment plants

OPTIMUM CONDITIONS AND SCHEME OF THE PROCESS

Injection of 3mL of the sample at pH=4

→ Load phase (Milli-Q water + 0,01% F.A.) is loading into the SPE cartridge at 2mL/min

While the chromatographic separation, cartridge 1 is cleaned and cartridge 2 is conditioned with the load phase

Sample loaded in the SPE is washed to eliminate interferences with 95% Milli-Q water / 5% MeOH

Analytes are eluted with the mobile phase (40% Water + 0,1% F.A./ 60% MeOH + 0,1% F.A.)

CONCLUSIONS
- All parameters affecting the process, such as loading phase, sample injection volume, sample pH and interference wash are optimized.
- The complete method, which involves extraction, preconcentration, separation and detection, takes place in 8.1 minutes.

REFERENCES
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[2] A. Garcia-Ac, P.A. Segura, C. Gagnon, S. Sauvé, J. Environ. Monit., 2009, 11, 830–838.