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# MONITORING OF 17 $\beta$ -ESTRADIOL IN RAW AND TREATED WATERS OF WASTEWATER TREATMENT PLANTS

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#### Introduction

Nowadays, there has been a growing concern regarding the possible consequences of exposure to estrogens through its direct or indirect consumption. The increasing utilization of estrogenic compounds, such as natural and synthetic estrogens, and pharmaceuticals has resulted in their continual occurrence in the aquatic environment. Monitoring the levels of estrogens is highly recommended due to its frequent detection in treated wastewaters of Wastewater Treatment Plants (WWTP's).

# **Experimental Methodology**

The present work is divided in two main experimental stages. First, an SPE/HPLC-UV experimental methodology is optimized to detect and quantify 17 $\beta$ -Estradiol (E2) present in aqueous samples. Secondly, the validation of the optimized experimental methodology is done by the monitoring of estradiol in wastewater influent and effluent samples from Bragança Wastewater Treatment Plant, in Portugal.



Figure 1: Bragança's WWTP

# Results and Discussion

Screening of HPLC-UV mobile phase

Table 1: HPLC-UV operating conditions analyzed

| INIOBILE PRASE COMPOSITION PR              |  |
|--|--|
| 50 ACN : 50 ULTRAPURE WATER 7.6            |  |
| 80 ACN : 20 ULTRAPURE WATER 5.9            |  |
| 100 ACN 5.2                                |  |
| 50 ACN : 50 ULTRAPURE WATER + 0.02 TFA 2.5 |  |
| 80 ACN : 20 ULTRAPURE WATER + 0.02 TFA 2.1 |  |
| 100 ACN + 0.005 TFA 1.7                    |  |
| 100 MET 5.8                                |  |
| 80 MET : 20 ULTRAPURE WATER 6.1            |  |
| 70 MET : 30 ULTRAPURE 6.4                  |  |
| 100 MET + 0.005 TFA 2.0                    |  |



Figure 2: HPLC-UV chromatographic pulses of estradiol using 8 different estradiol concentrations between 0.5 and 100 mg/L.

## SPE optimization using a 3-level Box-Behnken experimental design

**Table 2:** Experimental planning using the three-level Box-Behnkenexperimental design.

| FACTORS                        | LEVELS                  |
|--------------------------------|-------------------------|
| Sample Volume                  | 500, 1000 and 1500 (mL) |
| Sample pH                      | 2, 5 and 8              |
| Adsorbent drying time          | 10, 35 and 60 (min)     |
| Solvent composition in washing | 0, 5 and 10 (%)         |



#### Figure 3: Response surface for SPE procedures.

## Conclusion

Mobile phase of 100% methanol resulted in the best conditions to operate the HPLC-UV system, once it is the one that present a lower retention time. Regarding the SPE conditions, the maximum area (and also higher recovery of E2) is obtained using a sample with a pH value of 2, a sample volume of 500 mL, using 60 min for the adsorbent drying time and a 10% methanol added to ultrapure water in washing. Future work it is to analyze samples collected from Braganças's WWTP with methodology optimized.

#### References

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