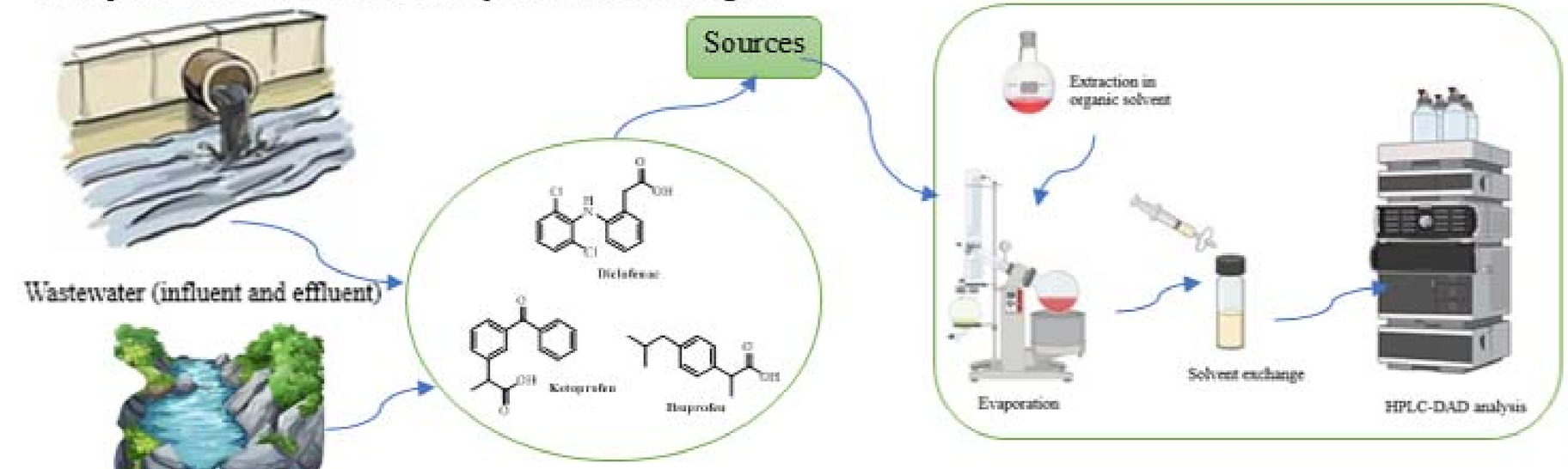
Environmental Monitoring of Pharmaceutical Pollutants: A Case Study on Anti-Inflammatories in the Wastewater and the Someș River, Cluj-Napoca

GÁL Emese[1], BRÉM Balázs[1], CAUACEAN Radu[2], BREHAR Marius Adrian[1,2], VERES-DANCIUT Thomas[1], PRODAN Amalia Ioana[1], BURCA Silvia[1], MIHALY Norbert Botond[1], TIMIS Elisabeta Cristina[1]
1. Babes-Bolyai University, Faculty of Chemistry and Chemical Engineering, Arany János 11, Cluj-Napoca Romania
2. S.C.Compania de Apa Somes S.A., Cluj-Napoca
emese.gal@ubbcluj.ro

ABSTRACT

The widespread detection and resistance of Non-Steroidal Anti-Inflammatory Drugs (NSAIDs) to conventional wastewater treatment poses a significant ecotoxicological risk. Our study employed a sensitive HPLC-DAD method to quantify key NSAIDs throughout the urban water cycle of Cluj-Napoca. We detected a substantial NSAID load in the WWTP influent and found significant residual concentrations in the effluent, directly contaminating the Someș River. This quantitative case study provides vital data for local risk assessment and necessitates the implementation of advanced tertiary treatment technologies.



Somes river before and after wastewater plant

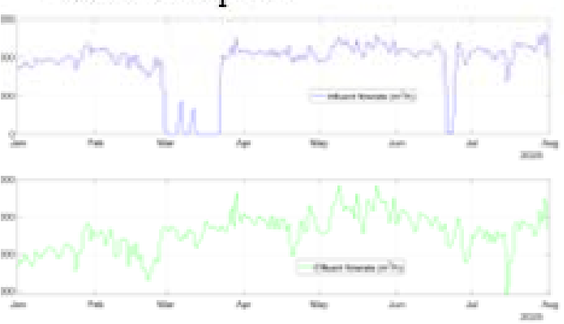


Fig. 1. Wastewater throughput at Someșeni

Sample preparation and Chromatographic conditions

500 mL of each wastewater sample were acidified at pH 3 with HCl 30%, extracted with dichloromethane (3x10ml), organic phase evaporated, and reconstituted in ACN:0.1% HCOOH in H_2O (55:45 v:v%). HPLC analyses were carried out with an Agilent 1200 equipment with a DAD detector. The studied NSAID's were separated on a column Luna C8 (150 x 4.6 mm, 5 µm) (Phenomenex, USA) with a flow rate of 0.8 mL/min. The injection volume was 20 µL. Isocratic elution was used ACN: 0.1% HCOOH in H_2O (55:45 v:v%). Ketoprofen – 256 nm, Diclofenac – 210 nm, and Ibuprofen – 230 nm.

Table 1. HPLC-DAD method validation results

Method validation, Limit of detection, Limit of quantification, correlation coefficient, per cent recovery for ibuprofen, ketoprofen, diclofenac					
Compound	LOD ($\mu g \cdot L^{-1}$)	LOQ ($\mu g \cdot L^{-1}$)	R^2	% Recovery ±%RSD	
				50 $\mu g \cdot L^{-1}$	5 $\mu g \cdot L^{-1}$
Ibuprofen	0.15	0.44	0.999	93 ± 1.6	96 ± 1.5
Ketoprofen	0.08	0.22	0.999	85 ± 2.5	89 ± 2.9
Diclofenac	0.13	0.41	0.991	89 ± 4.6	93 ± 3.4

Table 2. Obtained results during 1 months of sample measurements

Water samples Collected in Iune 20251062	Found concentration (min-max µg/L)		
	IBU	KET	DICL
Somes river Cluj-Napoca	>LOD- 152.3	>LOD- 49.52	0.52 - 141.9
Somes river Apahida	>LOD- 361.3	>LOD- 39.08	0.65 - 299.6
Wastewater influent	1.52 - 1243.5	0.80-217.97	0.25 - 969.6
Wastewater effluent	0.83-1062.5	3.84 - 125.7	>LOD- 266.9

Conclusion

In the samples collected the highest concentrations of the studied pharmaceuticals were found in wastewater influent. The samples from Their presence in wastewater effluent and Somes river shows their stability during the purification processes, the time and common uses of them. Our obtained results are comparable with other studies performed not only from samples from Cluj-Napoca but also performed worldwide.

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