May 08, 2024 Version 1

WATER PRODUCTION FOR AWARE (Organic Contaminants) V.1

DOI
dx.doi.org/10.17504/protocols.io.14egn67jql5d/v1

Antonio Cobelo[1]
[1]IIM-CSIC

AWARE Project
Horizon Europe 101084245

DOI: https://dx.doi.org/10.17504/protocols.io.14egn67jql5d/v1

Protocol Citation: Antonio Cobelo 2024. WATER PRODUCTION FOR AWARE (Organic Contaminants). **protocols.io https://dx.doi.org/10.17504/protocols.io.14egn67jql5d/v1**

Protocol status: Working
We use this protocol and it's working

Created: May 08, 2024

Last Modified: May 08, 2024

Protocol Integer ID: 99427

Keywords: water sampling, water processing, water analysis, waste water treatment, advanced tertiary treatment SOP, water production for aware, water production, standard operating procedure, procedure, organic contaminant, sop, optimization of advanced tertiary treatment, protocol, water, analytical control

Funders Acknowledgements:
Horizon Europe
Grant ID: 101084245

Abstract

The protocol summarises the procedures used for analytical control. The protocol describes the Standard Operating Procedure (SOP) for the optimization of advanced tertiary treatment of water, based on a comprehensive quality and risk assessment.

Guidelines

Directive 2008/105/EC of the European Parliament and of the Council of 16 December 2008 on environmental quality standards in the field of water policy.

Materials

	A	B	C	D	E	F	G	H
	Parameter	**V (mL) x R**	**S**	**Processing**	**Analytical method**	**Result**	**LOD / LOQ**	**Goal value**
	Organic contaminants	500 × 2	On ice	Solid-phase extraction	HPLC/MS-TOF	Presence/absence	-	Not present

Materials:

	A	B	C	D
		Becker		cartridges (OASIS HLB 200 mg)
	Pre-filteration	Buchner flask	**SPE**	vacuum pump
		PVDF 0.45 µm filters		graduated cylinder
		trap flask		becker
		vacuum pump		trap flask
				SPE vacuum manifold system

Troubleshooting

Safety warnings

Organic Contaminants:

1 The water production for AWARE main activities includes three stages – disinfection by ultraviolet C radiation (UVC), storage for 12:00:00 - 24:00:00 (according to water load and season) and ozonation. The water quality is monitored at these three stages, for the parameters indicated in Figure 1 below. 1d 12h

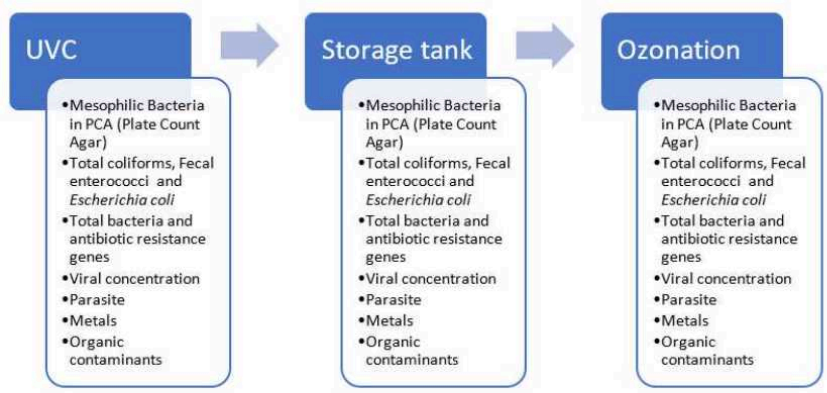

Figure 1. Treatment and storage of municipal treated wastewater used for integrated aquaponics and an indication of the comprehensive quality and risk assessment.

1.1 Sampling, Processing, and Analyses

9h

Water samples are collected (see Figure 2) and processed within a 06:00:00 interval, before being shipped for the partner responsible for the analyses (Table 1). In case no processing is needed, samples are frozen and stored at -80 °C within 03:00:00 .

For each sampling event, the date, day of the week and hour; the temperature and rain.
Sampling points, indicated in Figure 2 were designated from A to I:
- Influent of primary treatment (A)
- Influent of biological treatment (activated sludge) (B)
- Treated secondary effluent (C)
- Sand filter effluent (D)
- UVC effluent (E)
- Storage for reuse tank effluent (F)
- Ozonation effluent (1 dose, e.g., 5 mg O3) - MITO3X technology - (G)

- Effluent of the vacuum UV oxidation (VUV) (H)
- Effluent of reactive storage / Influent of the recirculation aquaculture system (RAS) (I) ù

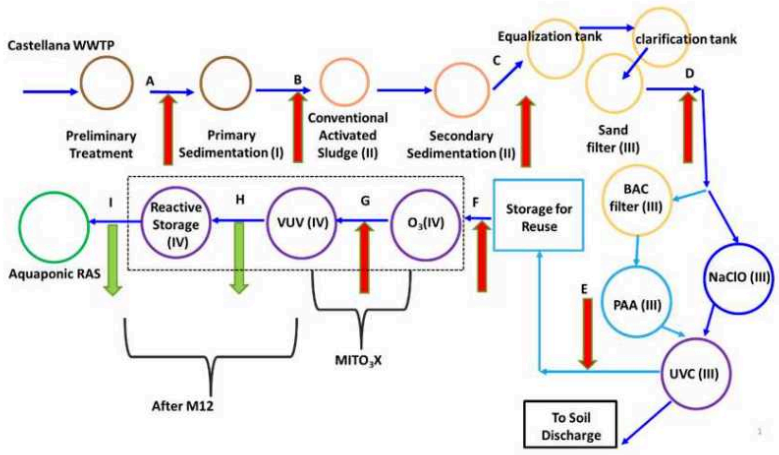

Figure 2. Diagram representing the wastewater treatment plant (WWTP), advanced treatment and sampling points.

Methods: The section below summarises the procedures used for analytical control – detailed protocols are annexed to this protocol.

1d 0h 30m

2 **Organic Contaminants:**

Analysis: Screening of Organic Contaminants in Water.

2.1 **Methods:** Solid-phase extraction

2.1 Sample filtration (**0.45um** PVDF).

2.2 Collect 200 mL of the filtered water sample (e.g. volumetric flask or beaker) and spike with 50 µL of an internal standard solution. Produce two 200 mL replicates per sample. Mix well after the internal standard is added.

2.3 Cleaning/Conditioning of the cartridges (OASIS HLB 200 mg)

3.1. Pass 5 mL of MeOH

3.2. Pass 5 mL of ultrapure water

2.4 Pass the 200 mL of sample

2.5 Rinse the volumetric flask or beaker that contained the sample with 2x 10 mL of ultrapure water, which are then passed through the cartridege

2.6 Drying the cartridge resin (e.g. N2 flow for 00:30:00) 30m

2.7 Store the cartridges in a freezer (-20 °C) 1d

All samples are to be processed in duplicate.

For the cartridge blanks (n=2): start with go to step #2.3 , and then spike directly on the cartridge the same amount of internal standard as for samples (50 µL mix de 0 mL). Then go directly to go to step #2.6 .
For the ultrapure water blanks (n=2), the same type of sampling flasks are filled with ultrapure water 24:00:00 before sampling. Then they are treated using the same protocols as for samples.

Elution of the loaded cartridges was carried out by gravity using 10 mL of methanol followed by 10 mL of dichloromethane. The eluates were concentrated approximately to ca. 0.5 mL using a Turbovap II concentrator (Zymark, Hopkinton, MA, USA), then to dryness under a gentle nitrogen stream and finally reconstituted in 500 µL of methanol and filtered with a 1ug mL GHP‰ 13 mm syringe filter (Pall Corporation). The extracts were injected in an Agilent 1290 LC coupled with and Agilent 550 QTOF system.

Parameters framed by Legal and Regulatory Requirements:

3 Using the EU Drinking Water Directive:

Organic contaminants - DIRECTIVE 2008/105/EC OF THE EUROPEAN PARLIAMENT AND THE
COUNCIL of 16 December 2008 on environmental quality standards in the field of water policy.

Protocol references

Montes, R.; Méndez, S.; Carro, N.; Cobas, J.; Alves, N.; Neuparth, T.; Santos, M.M.; Quintana, J.B.; Rodil, R. Screening of Contaminants of Emerging Concern in SurfaceWater and Wastewater.
Effluents, Assisted by the Persistency-Mobility-Toxicity Criteria.
Molecules 2022, 27, 3915. https://doi.org/10.3390/molecules27123915