# Development of a new microalgae-based wastewater treatment and reuse system with bioenergy production

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#### Abstract

A new wastewater treatment and reuse system, especially designed for small communities and isolated dwellings, is being developed in the frame of BioSolWaRe-LIFE Project (LIFE13 ENV/FR/000711). The system is based on the integration of an ecological process called "Bio-Solar Purification" (BSP) and the vacuum airlift (VAL). BSP combines both biological processes (microalgae cultivation in a tubular reactor) and the solar technology to remediate water. VAL technology enables water circulation and biomass harvesting, and combined with BSP, makes possible to reuse at least 80% of the reclaimed water. A first demo plant (Demo 1) has been built-up in Seville (Spain) with a nominal capacity of  $13 \text{ m}^3$ /day, approximately. The flow-sheet includes mechanical pretreatment, primary treatment (through Imhoff tank), the BSP/VAL unit, a VAL column for water clarification, and the post- treatment of the produced biomass through anaerobic digestion. The start-up hydraulic, treatment and digestion results of this Demo 1 are summarised in this study.

#### Keywords

Bio-solar purification; wastewater reuse; algae beneficial use; sustainable treatment; small populations

#### **INTRODUCTION**

Improving quantitative and qualitative water management as well as water treatment efficiency has become an increasing challenge in present times. The EEA State of Water report highlights worrying trends showing the increase and wider spread of water scarcity and stress, which is expected to affect in 2030 about half of EU river basins (EC, 2012). Water recycling and reuse is actually considered as the core of an integrated water management approach to save costs, recover materials and demonstrate environmental stewardship. Several eco-technologies at research and innovation stage, targeting on wastewater treatment, reuse and energy production, have been reviewed previously (García et al 2013). The BioSol Water Recycling LIFE project (www.life-biosol.eu) deals with this challenge by offering an innovative solution based on algae cultivation in closed systems (transparent tubular reactors) integrating Vacuum Air Lift technology, which will permit to save fresh water for reuse and prevent GHG releases. The project's objective is at least 80% of water recovery for reuse. BioSol Water Recycling project paves the way to a better water resources management in small and medium size communities and isolated dwellings where physico-chemical tertiary treatments are not competitive. Indeed, wastewater reuse allows water savings, water resources preservation, ensuring the decrease of hazardous substances release and preservation of aquatic ecosystems biodiversity. Moreover, small and medium size communities request simple but robust wastewater treatment and reuse systems, being the natural or extensive technologies, such as microalgae cultivation, actually appropriate for giving a sustainable long-term solution.

#### MATERIAL AND METHODS

Demo 1 (Figure 1) of BioSol Water Recycling LIFE Project has been built up in the R&D Experimental Centre of CENTA in Carrión de los Céspedes (Seville, Spain). The HelioPure ® BSP units have been developed by HPT (France) through pilots implemented in wastewater treatment plant. They consist in 16 transparent treatment tubes (125 mm diameter, 65 m length each, 128 m<sup>2</sup>), 4 feeding and collecting tanks connected to 2 Coldep<sup>®</sup> Vacuum Air-Lifts (VAL) columns for water circulation, O<sub>2</sub> stripping and CO<sub>2</sub> dissolution (1400/700 mm downward/upward column diameters, 6.2 m height) designed and provided by Coldep. The total system water volume is 37 m<sup>3</sup>.

Anaerobically pre-treated wastewater from Imhoff tank is fed to the BSP/VAL unit. After this process, water is conducted to a separation step ensured by a Coldep<sup>®</sup> VAL column (630/315 mm downward/upward column diameters, 4 m high), including the use of coagulating and flocculating agents, to obtain clarified water. The latter is reused for irrigation whereas the concentrated biomass (namely, microalgae) is released to a digestion unit provided by FCC AQUALIA. All the gathered information will be summarised in a Life Cycle Assessment (LCA), led by CENTA.

The results presented in this article deal with:

- Hydraulic trials: using electromagnetic flowmeter, the aim is to assess the performances of 1400/700 VAL columns for water circulation in BSP units.
- Individual batch treatment and separation trials for process optimization : The effect of circulating velocity in BSP tubes on treatment performances has been studied comparing the velocities of 0.17±0.02 m.s<sup>-1</sup> (7.4±0.9 m<sup>3</sup>.h<sup>-1</sup>) (BSPVAL-T-1) and 0.28±0.02 m.s<sup>-1</sup> (12.7±0.9m<sup>3</sup>.h<sup>-1</sup>) (BSPVAL-T-2). The comparison is based on kinetics assessment of several treatment indicators (BOD<sub>5</sub>, BOD<sub>5sol</sub>, COD, COD<sub>sol</sub>, TN, N-NH<sub>4</sub>, N-NO<sub>3</sub>, TP, P-PO<sub>4</sub>) that are monitored according to international standards related to water reuse in CENTA certified laboratory. Process parameters have been followed (pH, T, DO, chlorophyl concentration) at constant volume and sunlight intensity (direct and diffuse average intensity range in May ranging from 6 to 7 kWh.m<sup>-2</sup>.d<sup>-1</sup>) taking spot samples at 8 am and 8 pm every day during 6 days. Trials have been performed at higher temperature at highest velocity (BSPVAL-T-2 trials, mean value : 26.4±0.4°C, min/max : 15/35°C) compared to lowest velocity (BSPVAL-T-1, mean value : 20.5±0.8°C, min/max : 17/46°C) ; this temperature difference on the assessment of velocity effect will be discussed in the Results and Discussion section.
- Individual digestion trials: biogas from the digestion of the algal biomass harvested is monitored both quantitatively and qualitatively. At this stage, Biochemical Methane Potential (BMP) test has been implemented on micro-algae and mixed sludge conventional municipal WWTP (similar to CENTA WWTP). The use of BMP tests provides a relatively inexpensive, simple and repeatable method to make comparisons of the anaerobic digestibility and potential biogas potential between different substrates (Chynoweth et al, 1993). Moreover BMP tests can measure the residual organic material remaining after treatment that can still be used to convert to biogas and the non-degradable part remaining (Moody et al, 2009). Triplicate samples were studied for each substrate and mix used during the experiment. Tests were carried out in twelve 310-mL (useful volume) serum bottles capped with rubber septum sleeve stoppers supplied by ASM SOFT S.L. The reactors provide a data acquisition system for supervision and control supplied by SQL Server Express 2012 SP1. The reactors were placed in an incubator at 35 °C and shaking frequency of 200 rpm in stable conditions for a period of 21 days in a thermostatic chamber supplied by TC 135S AQUALYTIC and the orbital shaker was supplied by Phoenix Instrument Model: RS-OS 20.



Figure 1. Successive views of BSP/VAL (circulation), VAL separation and digestion processes

# **RESULTS AND DISCUSSION**

The whole BioSol Water Recycling facility has been first studied in batch mode in order to optimize operating procedures and controlled parameters on each component first.

# Hydraulic trials

Hydraulic trials have provided results on hydraulic performances of VAL columns in BSP/VAL system. When assessing water flow as a function of air flow, when varying number of working pumps from 1 to 3, the following figure has been drawn (Figure 2).

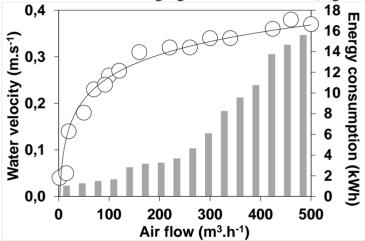


Figure 2. Hydraulic performances : water velocity as a function of air flow rates in VAL columns

Water velocity ranges from 0.04 to 0.38 m.s<sup>-1</sup> inside each tube (Figure 3), thus overcoming the 0.25 m.s<sup>-1</sup> settling threshold according to Stokes law while allowing the transport of organic matter particles. A velocity ranging from 0.3 to 0.5 m.s<sup>-1</sup> was reported as ensuring enough turbulence so that microalgae cells frequently move to the better illuminated peripheral zone and thus not starved of light for extended periods (Molina et al, 2001).

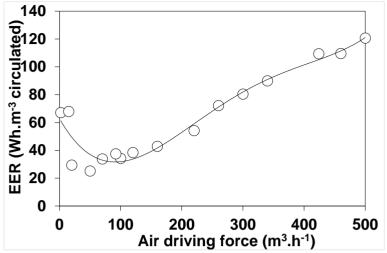


Figure 3. Energy consumption for water circulation in VAL columns

Among the existing circulating techniques, in optimized conditions, VAL columns require the lowest energy consumption per m<sup>3</sup> circulated water (up to 3 Wh.m<sup>-3</sup> without head losses), compared to axial pumps (10 Wh.m<sup>-3</sup>, considering a pump flow of 80 m3.h<sup>-1</sup> and installed power of 700 W) and centrifugal pumps (200 Wh.m<sup>-3</sup>, considering a Pedrollo pump flow of 250 m3.h<sup>-1</sup> and installed power of 47 kW) (Figure 3).

## Treatment and reuse batch trials

The study of the kinetics graphs and removal efficiencies (Table 1) indicates first that organic matter is degraded by bacteria/microalgae microorganisms and N-NH<sub>4</sub> is assimilated for protein production through photosynthetic phenomenon (Cai et al, 2013).

Moreover, this study shows that nutrient removal (g.m<sup>-3</sup>.d<sup>-1</sup>) is quicker at the lowest velocity when considering BOD<sub>5</sub>, soluble BOD<sub>5</sub>, COD, soluble COD whereas the reverse trend is drawn regarding TN, N-NH<sub>4</sub> and TP. On the other hand, initial concentration in Imhoff tank and the resulting one in BSP system is higher at lower velocity while chlorophylle concentration is initially 33% lower at lower velocity.

		Initial		Initial		Removal		Maximal volume and surface			
		concentration		concentration		efficiency		removal/production			
		in Imhoff tank		in BSP system		-		rates (from batch kinetics curves)			
		m	g.L <sup>-1</sup>	mg	.L <sup>-1</sup>	%	%	g.m <sup>-</sup>		g.m <sup>-2</sup> .d <sup>-</sup>	
								<sup>3</sup> .d <sup>-1</sup>	g.m <sup>-3</sup> .d <sup>-1</sup>	1	g.m <sup>-2</sup> .d <sup>-1</sup>
	Circulating	$7.4\pm$	$12.7\pm$	$7.4\pm$	12.7±	$7.4\pm$	12.7	$7.4\pm$	12.7±	7.4±	12.7±
	flow (m <sup>3</sup> .h <sup>-1</sup> )	0.9	0.9	0.9	0.9	0.9	±0.9	0.9	0.9	0.9	0.9
R	COD	437	277	498	371	68%	68%	222	158.2	23	16
Ε	COD <sub>sol</sub>	213	130	149	100	39%	6%	36	3.7	4	0
Μ	BOD <sub>5</sub>	260	160	190	120	79%	80%	187	102	19	10
0	BOD <sub>5.sol</sub>	115	55	60	18	93%	56%	160	13.5	16	1*
V	TN	68.2	36,7	42,7	34,7	40%	76%	9	21.4	1	2
Α	N-NH4	57.2	25,9	28,5	11,6	62%	96%	18.6	12.4	2	1
L	N-NO <sub>3</sub>	<5	<5	18,4	<5						
	TP	9	4,2	6,4	5,4	47%	61%	2.3	4.4	0.2	0.4
	P-PO <sub>4</sub>	6	2,6								
				Initial	Initial						
PROD				:	:1						
UCTI				913,24	555,95						
ON	Chloro-			Final :	Final :						
	phylle			537	51						

Table 1. Treatment performances as a function of circulating flow in BSP tubes

As a consequence of hydraulic results, the fact that wastewater entering BSP/VAL process has undergone a pretreatment removing 30% of organic load, in addition of mineral matter, may explain that velocity has not significant effect between 0.17 and 0.3 m.s<sup>-1</sup>.

Higher temperature at highest velocity (BSPVAL-T-1, mean value :  $20.5\pm0.8^{\circ}$ C, min/max :  $17/46^{\circ}$ C) compared to lowest velocity (BSPVAL-T-2, mean value :  $26.4\pm0.4^{\circ}$ C, min/max :  $15/35^{\circ}$ C) may increase the nutrient removal velocity, according to previous internal studies (HPT). An hypothesis would be that a temperature increase favours the metabolism of limiting nutrients (Hodaifa et al. 2010). Indeed, unfavourable conditions (i.e. high temperatures) increase the need of limiting nutrients (such as phosphorus and nitrogen). Similar results were obtained by Rhee and Gotham (2002).

On the contrary, the opposite trend may be drawn regarding soluble organic matter at similar removal percentages of total forms, since high temperature limit or even prevent microalgae growth and provoke population death. A 1000 mg/L decrease of chlorophylle concentration is measured at higher temperature (BSPVAL-T-2) compared to a 400 mg/L decrease at lower temperature (BSPVAL-T-1), leading to higher EPS production and increase of soluble organic matter forms, as well as different microalgae/bacteria population balance (Muñoz et al 2006).

So in our conditions, the controlling parameter is temperature compared to velocity and initial concentration to be treated, so it let suppose that circulating velocity has not a significant effect on organic matter treated inside BSP tubes in our conditions.

Theoretical surface removal rates have been estimated from volume removal rates considering  $13 \text{ m}^3$  treated volume and  $128 \text{ m}^2$  tube surface with HRT=24h (1 day) :

- TOC (organic form considered as mineralized carbon)~15 g.m<sup>-2</sup>.d-<sup>1</sup>.
- TN (organic form considered as mineralized nitrogen)~3 g.m<sup>-2</sup>.d<sup>-1</sup>.
- TP (organic form considered as mineralized phosphorus)~0.3 g.m<sup>-2</sup>.d<sup>-1</sup>.

Therefore, BSP/VAL rates are quite consistent with theoretical ones based on photosynthetic mass balance, considering that :

- BSP/VAL ones are based on maximal rates at initial treated concentrations;
- From mean organic carbon content in wastewaters, TOC has been estimated from COD values based on the following ratio : TOC=0.3-0.5 COD. So TOC would range from 4.6 to 11.5 g.m<sup>-2</sup>.d-<sup>1</sup>, with a mean value of 7.7±3.9 g.m<sup>-2</sup>.d-<sup>1</sup>.

Effective removal rates that will be used in order to design BSP/VAL facility, especially Demo 2, will be first assessed in continuous mode.

At this stage, these results at two different circulating velocities let confirm that daily (HRT=24h) treated volume may range between 13 m<sup>3</sup> and the overall capacity of 37 m<sup>3</sup> per day, given that the aim of BioSol Water Recycling unit is to allow wastewater reuse for agricultural purposes, instead of discharging it. This assumption will need to be confirmed following the study of the whole system performances including treatment and reuse, separation and biomass beneficial use together with irrigation.

## **Separation batch trials**

Following BSP/VAL treatment, it has been detected that microalgae biomass can be concentrated up to 2.2% Total Solids content in the VAL harvesting column, prior to the beneficial use for energy production by anaerobic digestion, while decreasing TSS concentration from 160 to 3 mg/L in wastewater (Figure 2) (98% efficiency). Further chemical and microbiological analysis need to be implemented regarding wastewater reuse for irrigation (Figure 4).

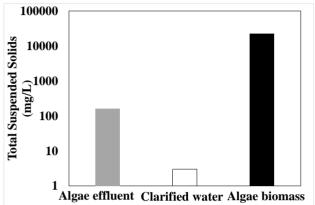


Figure 4. TSS contents at the inlet and outlet of separation process

On the other side, studies on laboratory scale of coagulating/flocculating processes applied on a new sample (compared to Table1) have provided first results regarding its contribution to disinfection process (Table 2). The quality level for wastewater reuse regarding French/Spanish regulations would allow reuse for crops eaten by humans and without direct contact of water with the plant. Indeed, focusing exclusively on reuse parameters, the results are : < 100 E. Coli FCUnits and Suspended Solids < 35 mg/L, COD < 125 mg/L. This indicative result will be confirmed through VAL separation trials in continuous mode.

Process	Quality level	Escherichia Coli	Suspended	Turbidity	COD
		(FCU/mL)	Solids		
French regulation	А	<250	<15		<60
_	В	<250	35		125
Spanish	2.1	<100	20	10	
regulation	2.2	<100	35	no limit	
<b>BSP/VAL</b> outlet		34000	75	23	222
Supernatant		<100	28±13	11±1	$122 \pm 3$
fraction					

Table 2. Disinfecting contribution of coagulating-flocculating processes

## **Digestion batch trials**

At this stage of the project, anaerobic digestion batch tests were performed at lab scale in order to assess the energetic beneficial use potential of microalgae biomass. Before continuous digestion trials, a batch digestion study has been conducted through BMP tests.

The goal was to evaluate different operational conditions such as biogas production, methane content, the biodegradability of the different substrates and the efficiency of the anaerobic process in order to compare the digestibility of mixed sludge from a conventional municipal WWTP (similar to CENTA WWTP) to a microalgae rich biomass (Table 3).

	Inoculum	Mixed	Inoculum	Micro-Algae
	Mixed Sludge	Sludge	Micro-Algae	
рН	7.82	6.47	8.00	6.86
COD <sub>total</sub> (mg O <sub>2</sub> /L)	$46160\pm2615$	$3371\pm75$	$29775 \pm 1285$	$54845 \pm 1600$
COD <sub>soluble</sub> (mgO <sub>2</sub> /L)	$370 \pm 30$	$70 \pm 10$	$1010\pm100$	$270 \pm 15$
Total Solids (mg/L)	$47520\pm5025$	$4005 \pm$	$27375\pm55$	$42535\pm270$
		270		
Mineral solids	$20185 \pm 1845$	$1780\pm95$	$8380 \pm 150$	$10040\pm205$
( <b>mg/L</b> )				
Volatile Solids	$27335\pm3185$	2225 ±	$18995 \pm 115$	$32500\pm95$
(mg/L)		230		
Ammonium (mg/L)	660	31	940	136
NTK (mg/L)	3483	224	3046	3153
Sulphates (mg/L)	47.1	147.6	22.2	96.7

Table 3 Initial Characterization of the substrates and inoculums

As shown in Table 4, the biogas production from microalgae biomethanization presented a richer methane composition than mixed sludge. Volatile Solids (VS) have been assessed through APHA 2540 method. The biogas composition (CH<sub>4</sub>, CO<sub>2</sub>, H<sub>2</sub>S and O<sub>2</sub>) was analysed with a portable gas analyser COMBIMASS® (BINDER group).

Table 4. Biogas co	mposition
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	CH4(%)	CO <sub>2</sub> (%)	CH4/g VS (mL)
Mixed Sludge	$58.4\pm6.4$	$41.6\pm6.4$	153
Microalgae	$80.5\pm2.1$	$19.5 \pm 2.1$	186

Table 4 indicates that the accumulative methane yield is 18% lower with mixed sludge digestion. In our study, with TS concentration of 4 and 42 g/kg for mixed sludge and microalgae, respectively, methane productivity remains similar (153 to 186 mLCH4/gVS algae) while S/I ratio is much lower with mixed sludge (0.08) compared to microalgae (1.5). Other study varying TS concentration from 3 to 20 g/kg reported methane productivity from 150 to 400 mLCH4/gVS at S/I TS ratio of 0.5 (Alzate et al 2012). So in our study, S/I ratio does not seem to get a significant effect or its effect may interfer with other parameter effect.

Mixed sludge showed a sigmoidal curve of cumulative methane production, which means that the hydrolysis phase is the limiting step of the process. Such behaviour would be associated to the inoculum composition, having lower hydrolytic potential the one used in the anaerobic digestion of mixed sludge. Indeed, normally BMP are comparable only with similar inoculum.

Another important factor could be related to the sulphate concentration (Table 3) since its content was higher in mixed sludge (147.6 mg.L<sup>-1</sup>) compared to microalgae (96.7 mg.L<sup>-1</sup>). Sulphate reducing bacteria compete with methanogenic bacteria for acetate and hydrogen, especially at sulphate concentrations higher than 150 mg/L when it becomes extremely toxic to methanogenic bacteria. Two types of inhibition are related to the sulphate reduction process:

- a) The first inhibition is due to competition for common organic and inorganic substrates from sulphate reducing bacteria (SRB), which suppresses methane production (Harada et al., 1994)
- b) The second inhibition, results from the toxicity of sulphide to various bacteria groups (Anderson et al., 1982; Oude Elferink et al., 1994; Colleran et al., 1995; Colleran et al., 1998).

Anaerobic biodegradability was reported by comparing cumulative methane yield (mL CH<sub>4</sub>/g COD) obtained from each test against the theoretical maximum yield resulting from the complete degradation of this substrate (350 mL CH<sub>4</sub>/g COD). Both substrates presented similar results, being the biodegradability values slightly higher for microalgae (Table 3).

Table 5. Biodegradability values

	Biodegradability
Mixed	$28.8 \pm 7.4$
Sludge	20.0 ± 7.4
Micro-Algae	$31.5 \pm 2.9$

## CONCLUSIONS

These batch trials on each component of BioSol Water Recycling facility let optimize operational conditions regarding treatment and reuse efficiencies :

- > Hydraulic trials allowed determining sizing diagrams and optimal working range
- > Treatment and separation trials have shown degradation kinetics regarding targeted parameters for water treatment.
- Digestion trials showed the higher methane production yield of microalgae compared to mixed sludge, while presenting similar biodegradabilities.
- LCA analysis (results not enclosed) on design and construction has allowed identifying alternative materials in order to minimize energy consumption and carbon footprint. Process design and operation is being optimized so detailed carbon and energy balances will be provided in further studies.

In addition, this study provides interesting perspectives consisting in operating the process at low velocity (0.15 m.s<sup>-1</sup>) or even lower in order to reach similar performances at lower energy consumption. Treatment and reuse performances, carbon and energy balances will be studied by optimizing together Hydraulic Retention Times of BSP/VAL and VAL separation processes. These performances will be discussed considering existing wastewater treatment regulations (2000/60/CE, 91/271/EC) and the following regulations for wastewater treatment and reuse, with the view to develop a competitive solution meeting the requirements of agricultural irrigation.

The aim of this study is both preparing trials in continuous mode and optimizing the design of the whole plant (Demo 2), increasing tube length up to 100 m in Almeria WWTP. This final unit able to treat and reuse 50 m<sup>3</sup>.d<sup>-1</sup> of wastewater will be operated in the WWT plant of Almeria by the end of 2016. Indeed solution dedicated to wastewater treatment and reuse for agriculture, such as BioSol Water Recycling system, target at meeting treatment and reuse needs resulting from high local hydric stress and high demand for intensive farming in greenhouses.

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