Circular economy applied to the treatment of table olives brines based on solar evaporation

LIFE SOLIEVA: Main results from membrane stage and advanced solar evaporation

17th June 2022

Joan Tarragona, Eurecat

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WHY SOLIEVA?

Table olive world production (prev. 2018/2019, IOC 2018)

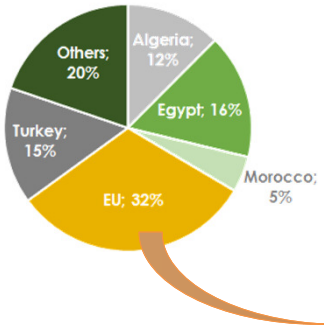
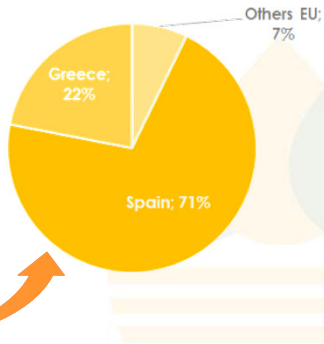


Table olive EU production (prev. 2018/2019, IOC 2018)



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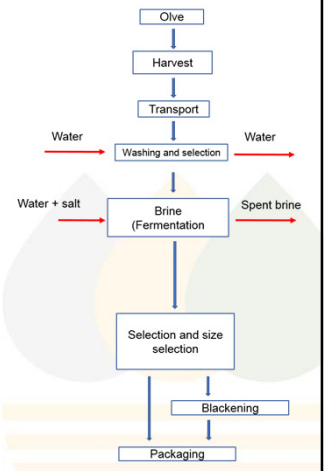
the LIFE+ SOLIEVA project has received funding from the LIFE programme of the European Union

WHY SOLIEVA?

Table Olive Production Wastewater (TOPWW)

- ✓ Complex composition
- ✓ Seasonality

SPAIN			
	Pickling industries	**Packaging Industries**	**Total**
Yearly Production (tn/year)	650,000	264,000	914,000
Generated wastewater (L/kg of produced olives)	Green style: 2 Black style: 4 Average: 1.5	0.7 [0.6-0.8][1]	-
Volume generated yearly (m^3/year)	975,000	184,800	1,159,800



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WHY SOLIEVA?

Table Olive Production Wastewater (TOPWW)

- ✓ Complex composition: High salinity: NaOH, NaCl, Organic Matter (Polyphenols)
- ✓ Biological conventional treatments not effective

Parameters	Lye		Washing waters		Brine	
	Average	Range	Average	Range	Average	Range
pH	12.1	9.5-13.2	10.0	7.2-11.5	4.0	3.6-4.6
Acidity (g of lactic acid/L)	-	-	-	-	8.0	3.5-15.0
Electrical conductivity(mS/cm)	12.1	11.1-13.0	10.2	-	81.4	53.1-94.2
COD (g O_2/L)	18.8	9.4-35.0	16.1	0.3-35.0	15.9	6.8-26.0
BOD (g O_2/L)	9.5	3.1-20.0	11.0	0.1-21.0	10.6	2.2-20.0
Dissolved organic solids (g/L)	21.9	13.1-30.0	24.2	19.1-30.0	18.8	13.6-25.2
Dissolved inorganic solids (g/L)	21.9	15.4-35.0	20.0	6.8-54.3	73.9	20.9-110.0
Total suspended solids (g/L)	2.03	0.10-3.42	0.07	0.03-0.10	0.81	0.08-2.00
Total phenolic solids (g/L)	1.78	0.21-4.00	2.32	0.45-4.00	2.78	0.18-6.00
Sugars (g/L)	6.6	4.9-9.0	6.4	4.7-9.0	-	-
Total Kjeldahl nitrogen (g/L)	0.58	0.5-.075	-	-	0.32	0.27-0.36
NaOH (g/L)	9.0	6.9-11.0	1.5	0.9-2.0	-	-
NaCl (g/L)	0.0005	0-0.0010	0.0005	0-0.0010	67.8	52.0-90.0
Cl^- (g/L)	0.32	0.00-0.60	0.30	0.00-0.60	49.1	36.4-62.7

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WHY SOLIEVA?

Current management

Evaporation ponds

- ✓ High land occupation
- ✓ Very low efficiency in resources use, losing resources like water, salt and polyphenols
- ✓ Uncontrolled sludge accumulation
- ✓ Raise of spillage risk due to overflows by high intensity rain

Conventional forced evaporators: ✓ High energy consumption and CO2 emissions

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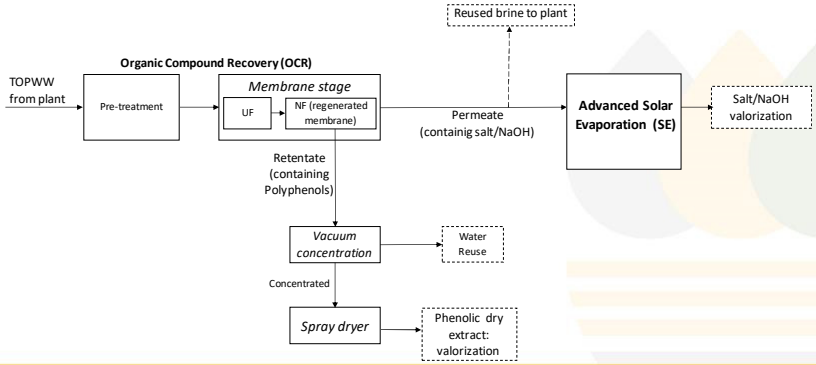
SOLIEVA OBJECTIVE

Demonstrate the technical, environmental and economic feasibility of the technology based on membrane, concentrator vacuum and spry dryer (OCR) and advanced solar evaporation (ASE) to face the environmental challenges of the table olive sector (TO) in the treatment of the table olive processing wastewater (TOPWW) and update the environmental legislation related to use of water.



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SOLIEVA OBJECTIVE

- ✓ To demonstrate de innovative treatment scheme at pilot scale
- ✓ To improve the resource efficiency of TO sector with the reduction in the water consumption and TOPWW generation and the reuse of salt and caustic soda in the TO production and into de market
- ✓ To define valorisation pathways of polyphenols recovered for the formulation of functional foods and subproducts.
- ✓ Environmental, social and economic assessment from the uptake of SOLIEVA technology, and define the replication and transferability plan in TO regions and other agrofood industries.
- ✓ Scale up of SOLIEVA and definition of a business plan.
- ✓ Provide recommendations for the update of wastewater treatment legislation.

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SOLIEVA APPROACH

Development of innovative treatment scheme that enable the recovery of valuable resources from TOPWW while avoiding the environmental impact of the current management strategies.

INNOVATIVE TECHNOLOGIES

Separation of organic matter by using regenerated membranes

Zero Liquid Discharge by Advanced Solar Evaporation

RESOURCE EFFICIENCY

Resources recovery: salt, NaOH, water

Recovery and valorization of polyphenols

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DEMONSTRATION SITE

OLEAND – Cooperativa Nuestra Señora de las Virtudes
La Puebla de Cazalla (Sevilla)

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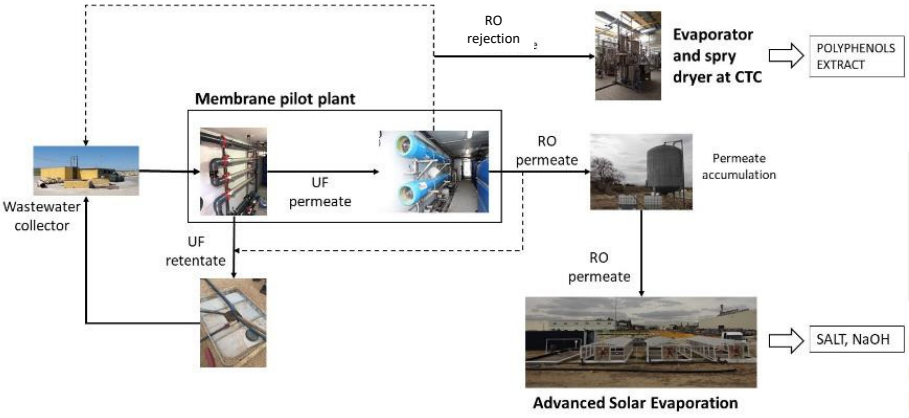
PILOT PLANT



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Membrane stage

Ultrafiltration

RO/NF regenerated membranes

SCADA: Monitoring

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Membrane stage

Production of 2 types of membranes for the pilot:

	Regeneration dosis [ppm·h]	NaCl rejection [%]	Permeability [LMH/bar]	
Membrane type I (1)	56,000	64,1	8,33	**Membranes with ~ 60% NaCl rejection**
Membrane type I (2)	46,000	59	9,65	
Membrane type II (1)	32,000	71,2	8,73	**Membranes with ~ 75% NaCl rejection**
Membrane type II (2)	22,000	76,3	8,17	

*Rejection and permeability based on NaCl test: 15% od recovery, 25 ºC, 2000 ppm NaCl, 16 bar

Operation from February 2020 to December 2021.

Near 1000 m³ have been treated with the pilot plant using both type of membranes

Membrane performance has been evaluated **taking daily samples**

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Membrane stage

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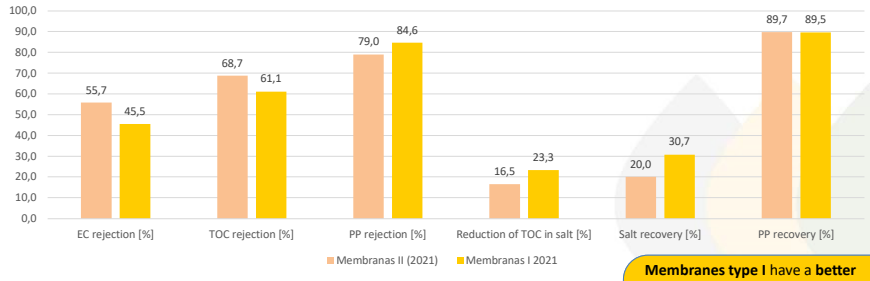
Membrane stage

Results for the operation of regenerated RO membranes (average values):



	Operation Pressure [bar]*	Water recovery [%]*
Membrane type I	41	61
Membrane type II	42	54

*Average values for sample points

Membranes type I have a **better performance** in terms of:

- ✓ Salt recovery in permeate
- ✓ Reduction of TOC/salt rate
- ✓ PP recovery (same as type II)
- ✓ Water recovery (brine)

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Advanced Solar Evaporation

How evaporation is intensified?

Advanced control based on phisical model

Control of the air and water conditions

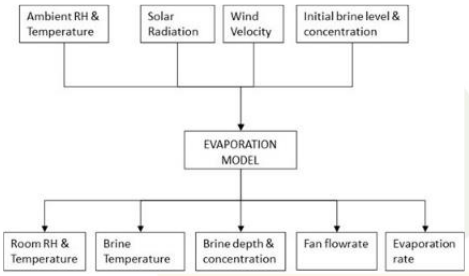
Filling control (temperature increase)

Optimization of the air circulation

Advanced design

Heat losses minimization

To avoid volume increase due to rainfall
Maximized solar radiation collection



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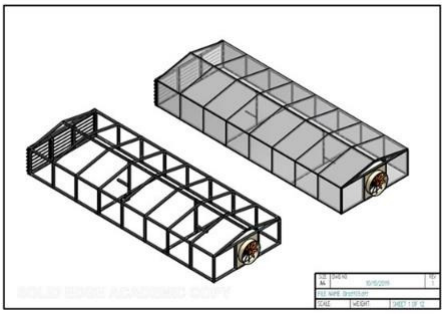
Advanced Solar Evaporation



•**Filling system** – Brine pre-treatment

It consists of the components for the dosage and supply to the evaporation pool.

•**Evaporation pond** - Evaporation system

formed by the main components of the pool and the envelope, where the evaporation of the water is carried out.

•**Ventilation system**

It consists in the extraction of humid air through a fan and a low pressure drop persian blinds.

•**Maximization of solar radiation collection.** Greenhouse effect, solar absorbent pool coating

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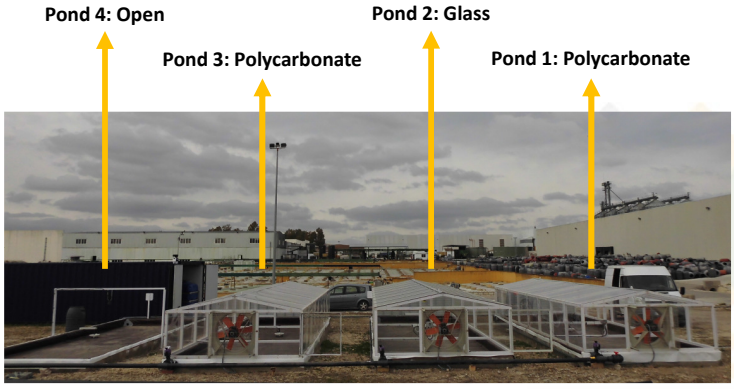
Advanced Solar Evaporation



25m2 each pond

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Advanced Solar Evaporation

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Ventilation system
Activated and deactivated by advanced control

Fan: extracts air from inside producing a pressure drop inside the pond

Blinds opened by pressure drop inside produced by fan

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Advanced Solar Evaporation

Brine level, brine temperature, conductivity
Temp. and Humidity inside and outdoor
Wind, rainfall

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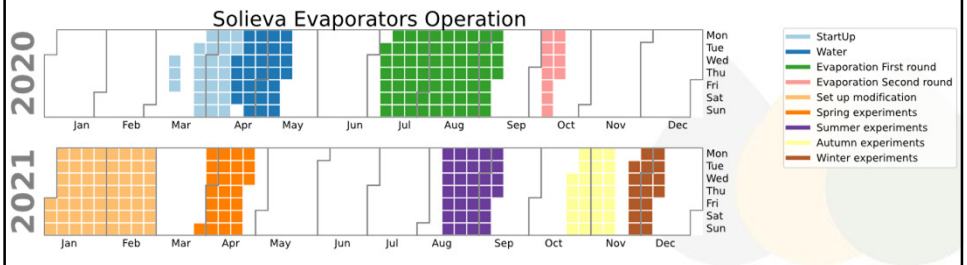
Advanced Solar Evaporation



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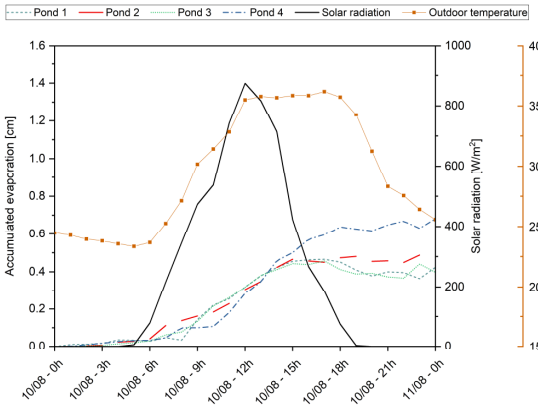
Advanced Solar Evaporation

Summer experiments



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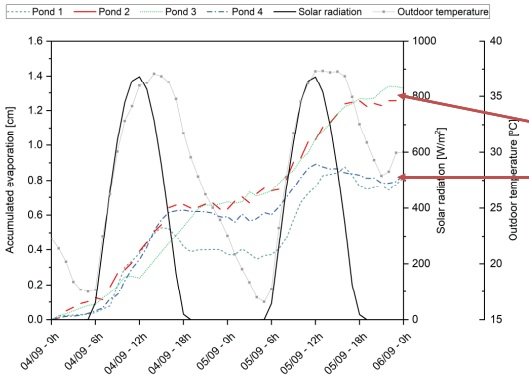
Summer experiments



Ventilation intensified

ASE

Open pond

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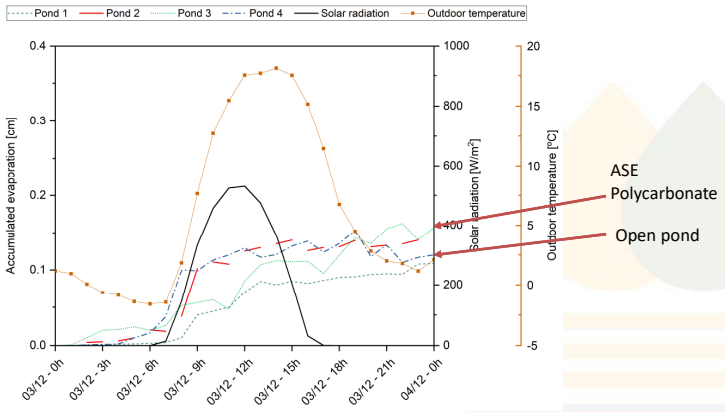
Advanced Solar Evaporation

Winter experiments



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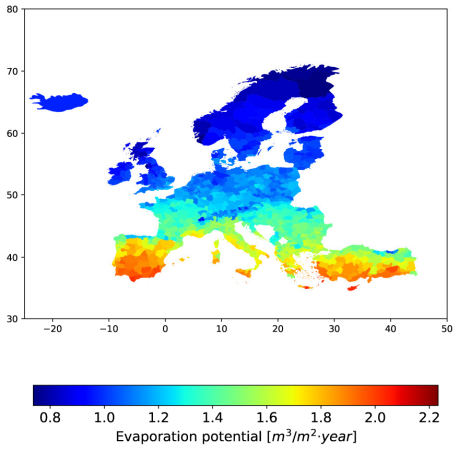
Advanced Solar Evaporation

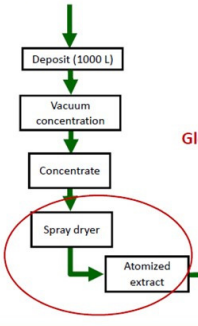
- The **hot weather conditions** of La Puebla de Cazalla promote the increasing of the evaporation rate of the open ponds
- The **rain** has a significant negative impact upon the performance of the open ponds
- The **ASE is less sensitive than open ponds to the bad weather conditions** (low solar radiation, low temperature, and high humidity)
- Although the high evaporation ratio of the open pond during the summer season, the ASE can overcome its performance by means of intense air renewable

Advanced Solar Evaporation



Polyphenols recovery

TOPWW - from plant
Retentate



Initial °Brix of concentrate: 5-9; Media °Brix: 7
Work criteria: °Brix ≤ 16 (Maximum value to work Spray Dryer)
Maximum yield of the vacuum concentrator: 82,8% (~40 °Brix)
Work yield of vacuum concentrator: 56.25% (~16 °Brix)

Global process yield: 92.5%

Atomized extract

Atomized extract
96,8 % Dry Matter
72 % of Salt, 28 % of MO
Total polyphenols: 6 – 8 %

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Polyphenols recovery

Atomized extract → Recovery and purification of polyphenols Extraction with ethanoic distillation → Polyphenols extract → Stabilisation (Maltodextrin) → Lyophilization → Polyphenol extract

96,8 % MS
72 % of Salt, 28 % of MO
Total polyphenols: 6 – 8 %

96,8 % MS
24,6% of Salt, 75,4% of MO
Total polyphenols: ~13 - 16 %

Determinación	Resultados
Ácidos grasos Saturados g/100g	0.3
Azúcares totales g/100g	28.2
Cenizas totales g/100g	13.9
Cloruro sódico g/100g	10.4
Grasa g/100g	0.6
Hidratos de Carbono g/100g	81.5
Humedad g/100g	2.5
Proteínas g/100g	1.5
Valor energético (kcal) Kcal/100g	337
Valor energético (kJ) kJ/100g	1433
Hidroxitirosol mg/kg	72112
Tirosol mg/kg	2291
Polifenoles totales mg/kg	69.600

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Polyphenols valorization pathways

NEW FOOD FORMULATIONS

¿?

1. Ingredients
2. Refrigerated storage products
   (Shelf life study)

8 NEW PRODUCTS

CTC Centro Tecnológico Nacional de la Conserva y Alimentación

ZUCCHINI CREAM
LETTUCE GAZPACHO
PEPPER STUFFED OLIVES
HUMMUS
TOMATO JUICE

CITOLIVA

BISCUITS
OLIVE PATE
ICE CREAM

1. **Formulation**
   (control, 0,5%-2% polyphenol purified ext.)
2. **Nutritional Analysis**
3. **Sensorial Analysis**
4. **Shelf life study**

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Polyphenols valorization pathways

NEW FOOD FORMULATIONS
ZUCCHINI CREAM

INGREDIENTS g/100g	CONTROL	Ext 0.5%	Ext 1%
ZUCCHINI	28.22	28.22	28.22
ONION	11.45	11.45	11.27
LEEK	6.45	6.45	6.45
POTATO	6.45	6.45	6.45
OLIVE OIL	1.61	1.61	1.61
WATER	45.18	45.08	45.00
PURIFIED EXTRACT	0.00	0.50	1.00
SALT	0.64	0.24	0.00

- WASHING AND ADAPTATION OF RAW MATERIALS — INGREDIENTS: Potato, zucchini, Onion, Leek
- PEELED
- URSCHELL CUTTER 10X10 mm — INGREDIENTS: Potato, zucchini, Onion, Leek, Olive oil, salt and Water
- COOK 30 MIN
- CRUSHED
- ADDITION OF PURIFIED EXTRACT SOLIEVA — Control, 0.5%, 1%
- HOMOGENIZED
- PACKING
- REFRIGERATION STORE

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Polyphenols valorization pathways

Doughs: DARK CHOCOLATE BISCUITS WITH WHOLE FLOUR ("DIGESTIVE" TYPE)

Ingredients:

- 100% whole wheat spelled flour
- Baking powder
- Butter
- Powdered sugar
- Milk
- Dark chocolate (85% cocoa)
- Polyphenol extract provided by CTC:
  - 1%
  - 2%

Polyphenols valorization pathways

FOOD	Shelf life (microbiological values)	Shelf life (organoleptic values)	pH and moisture
Tomato juice	=	≅	Not evaluated
Zucchini cream	+	-	Not evaluated
Lettuce gazpacho	+	-	Not evaluated
Pepper stuffed olives	-	≅	Not evaluated
Hummus	+	-	Not evaluated
«Digestive» biscuits	Not evaluated	+	Not evaluated
Olive pate	-	-	≅
Ice cream	≅	=	≅

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Polyphenols valorization pathways

❑ **General conclusions:**

- The polyphenols dry extract presents characteristics and technological capacities **suitable for use in the food industry** for the elaboration of new food formulations.
- The contribution of **color and flavor** to food is significant.
- Shelf life study shows the **antimicrobial capacity** of the purified extract for liquid food (creams, juices and sauces). In food containing high starting microorganism concentration is not efficient reducing the microbial load.
- **Organoleptic evolution** shows the ability of the purified extract to extend the self life in biscuits but not in food not cooked (ice cream, olive pate)
- The organoleptic properties of the polyphenol extract make it necessary to **select the ingredients** for the formulation of new foods.

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ECONOMIC FEASIBILITY

	Conventional (Forced evaporation)	SOLIEVA
TOTAL-capital good	0,19 €/m3	1,62 €/m3
TOTAL-operation	12,58 €/m3	6,99 €/m3
TOTAL-products		500 €/m3
TOTAL COSTS(€/m3)	12,77€/m3	8,61 €/m3

- ✓ ***Economic feasibility***: SOLIEVA technology allows lowering operating costs and obtaining significant economic benefits.

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MAIN CONCLUSIONS

- ✓ Technical and economical feasibility of SOLIEVA solution has been demonstrated
- ✓ Legal issues to be considered for the acceptance of polyphenols extract as additive.
- ✓ Segregation of brine from another wastewater is recommended.
- ✓ Depending on the membrane used different streams quality. Water for irrigation purposes can be produced.
- ✓ Authorization from the sanitary inspection service from the administration for permeate reuse
- ✓ **Decision Support Tool** to provide information about technical, environmental and economic considerations for the uptake of SOLEIVA technology.

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