Application of NaCl-plant extracts to decrease the costs of microfiltration for winery wastewater treatment

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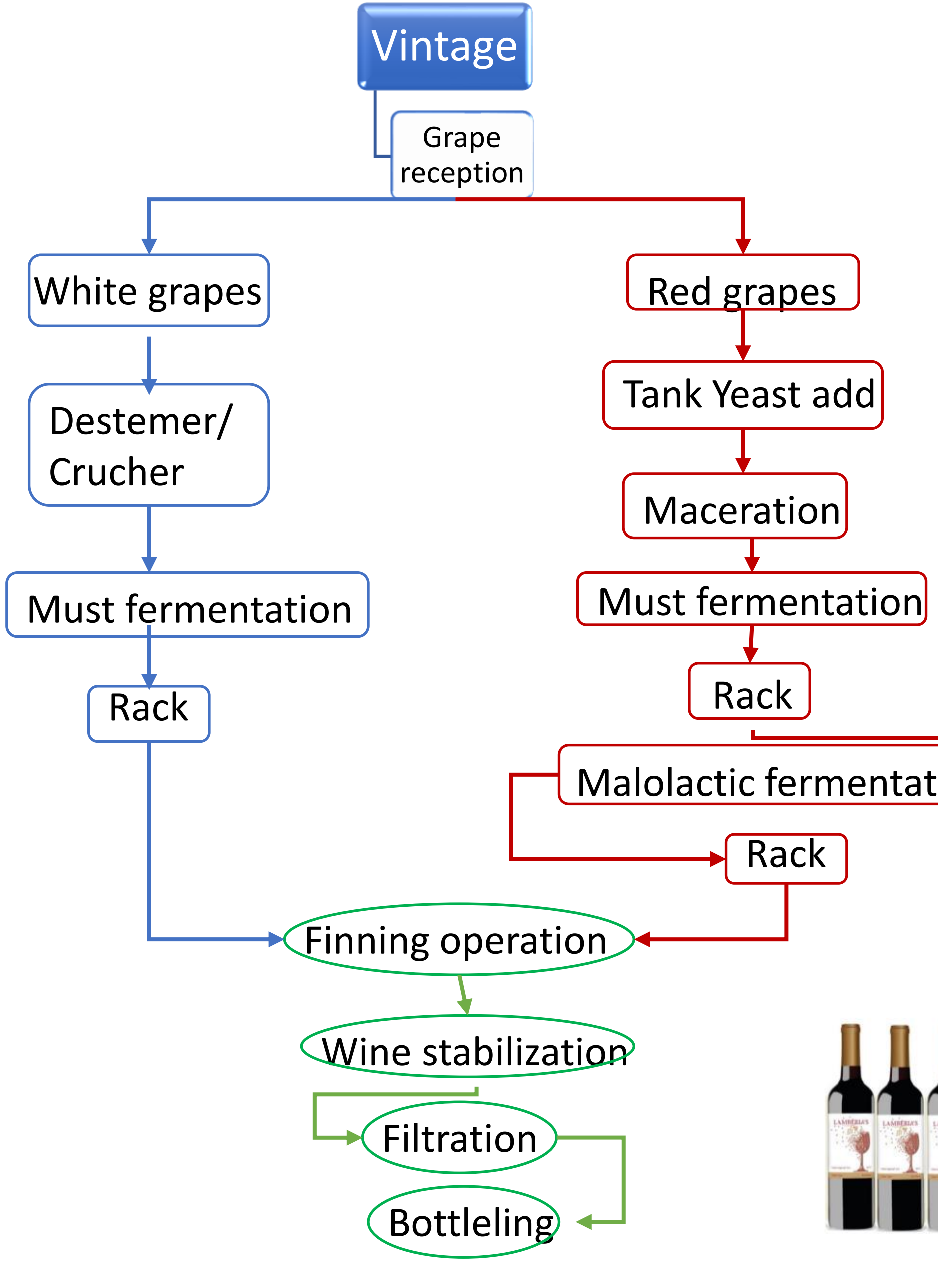




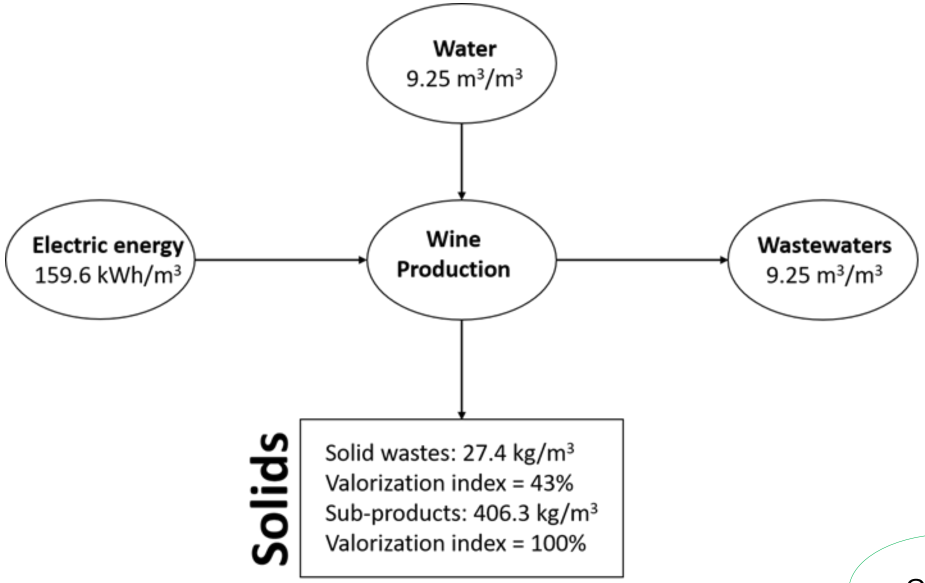




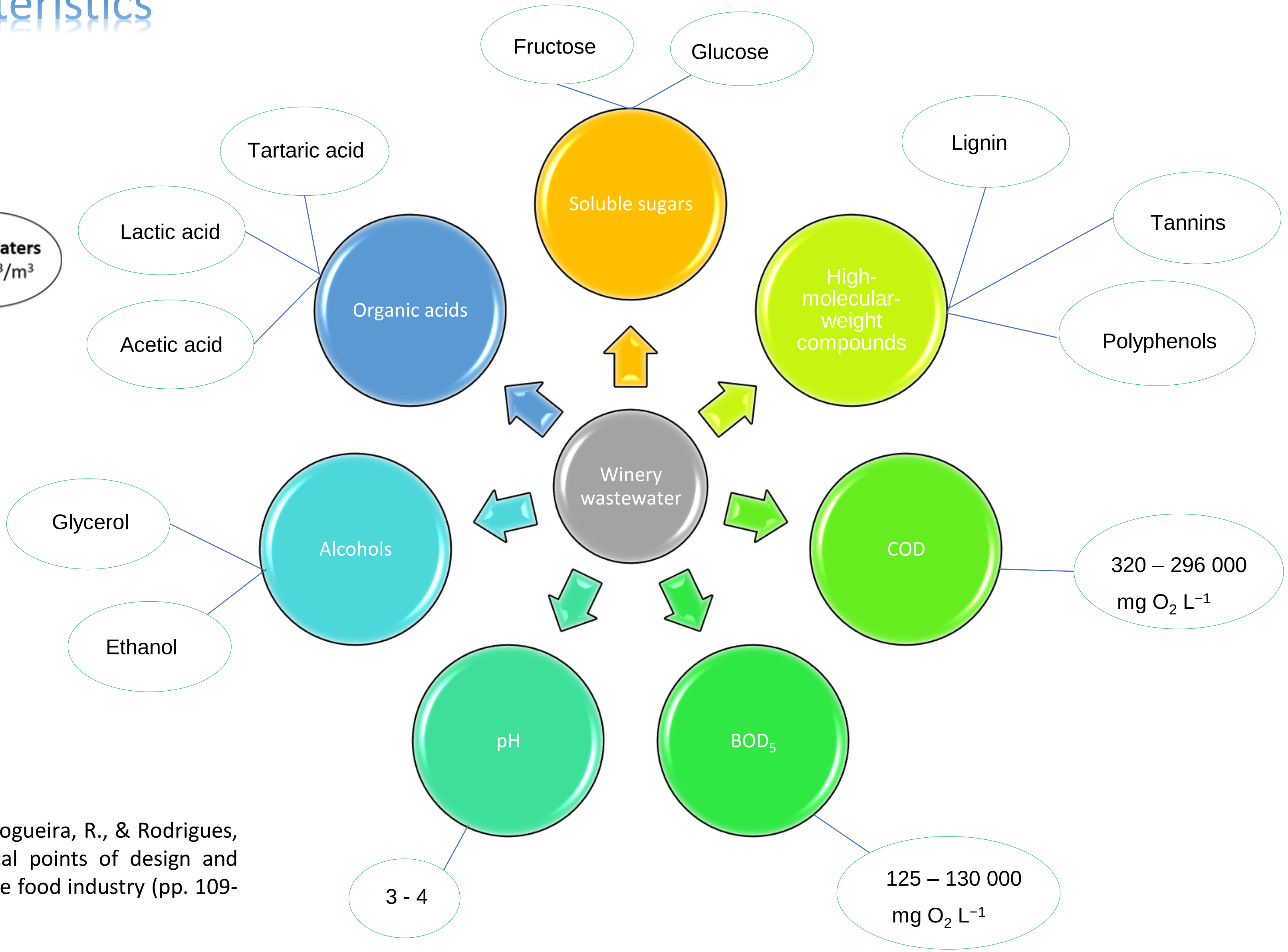



- In the vintage, the grapes are collected
- In the grape reception, the grapes are selected and separated
- In white wines the grapes are crushed and the must is fermented
- In red wines, the grapes are marecerated and the must is fermented with the grapes
- After must fermentation and wine stabilization, the wine is filtrated
- Finally, the wine is bottled

Winery wastewater main characteristics



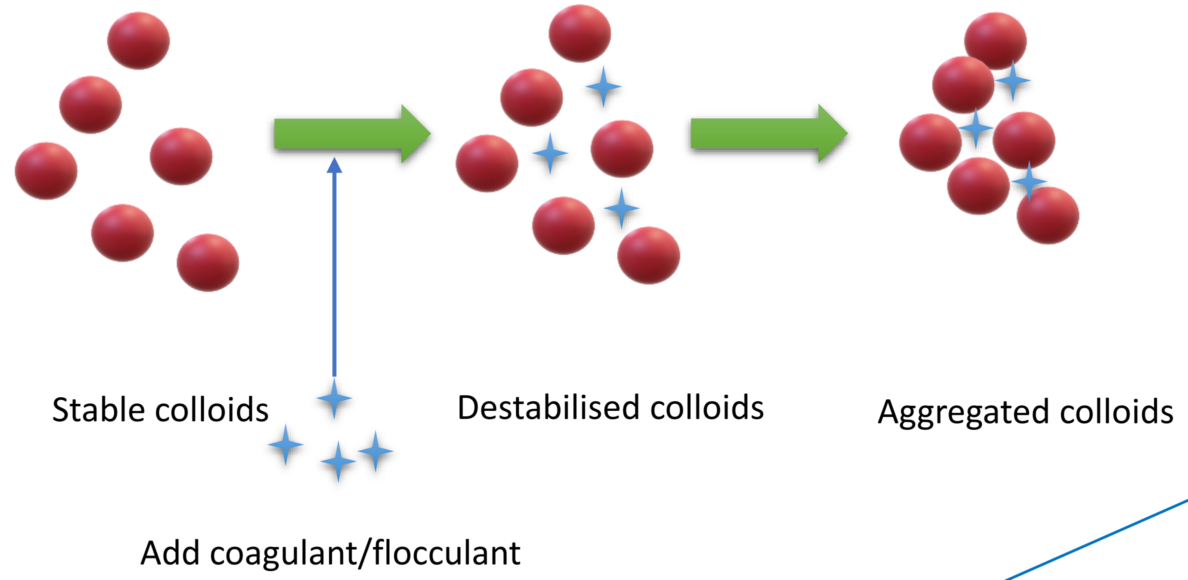
Mass balance applied to ACPB winery representing specific values, i.e., values per cubic meter of produced wine. Losses of water by evaporation were neglected [1].



[1] Brito, A. G., Peixoto, J., Oliveira, J. M., Oliveira, J. A., Costa, C., Nogueira, R., & Rodrigues, A. (2007). Brewery and winery wastewater treatment: some focal points of design and operation. In Utilization of by-products and treatment of waste in the food industry (pp. 109-131). Springer, Boston, MA.

Physical-chemical treatments of wastewater

Coagulation-flocculation-decantation (CFD)



- Hydrolysable metal salts (mainly, **aluminum** and **iron**)

  Most used on waster treatment

Disadvantages

Aluminum

- Dialysis encephalopathy
- Alzheimer's disease

Iron

- Generally corrosive
- Strongly dependent on the pH
- The leach cannot be recycled

Plant species colected during this work, for the development of **Na-Cl plant extracts**

Acacia dealbata Link (pollen)

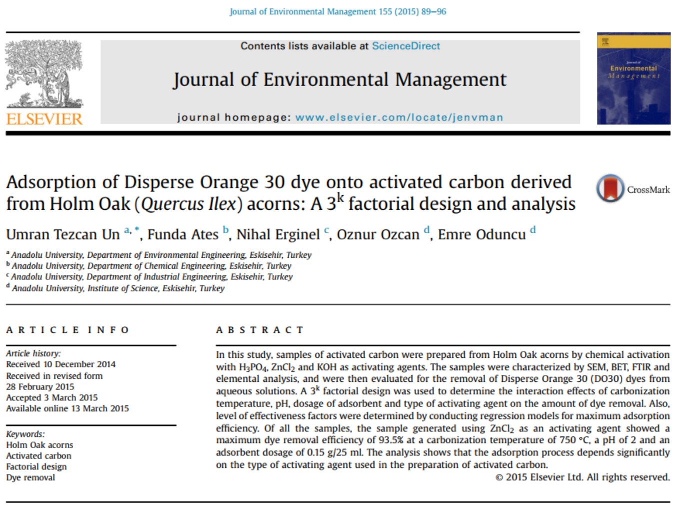
Chelidonium majus L. (seeds)

Tanacetum vulgare L. (seeds)

Daucus carota L. (seeds)

Vitis vinífera L. (rachis)

Works performed with plant based coagulants



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Adsorption of Disperse Orange 30 dye onto activated carbon derived from Holm Oak (*Quercus Ilex*) acorns: A 3^k factorial design and analysis

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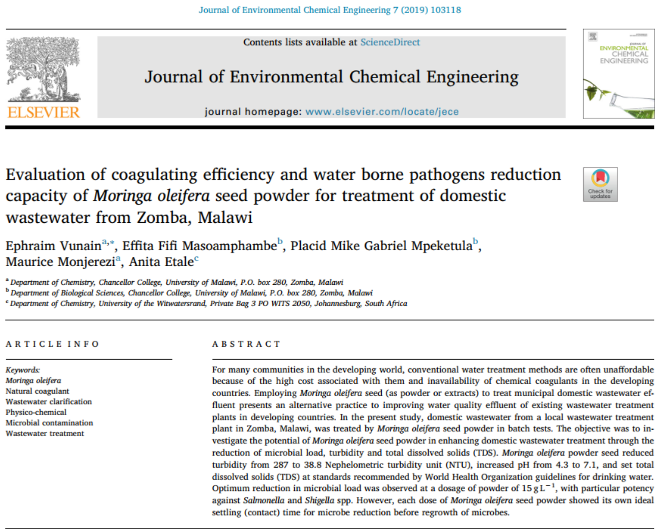
ABSTRACT

In this study, samples of activated carbon were prepared from Holm Oak acorns by chemical activation with H_3PO_4, $ZnCl_2$ and KOH as activating agents. The samples were characterized by SEM, BET, FTIR and elemental analysis, and were then evaluated for the removal of Disperse Orange 30 (DO30) dyes from aqueous solutions. A 3^k factorial design was used to determine the interaction effects of carbonization temperature, pH, dosage of adsorbent and type of activating agent on the amount of dye removal. Also, level of effectiveness factors were determined by conducting regression models for maximum adsorption efficiency. Of all the samples, the sample generated using $ZnCl_2$ as an activating agent showed a maximum dye removal efficiency of 93.5% at a carbonization temperature of 750 °C, a pH of 2 and an adsorbent dosage of 0.15 g/25 ml. The analysis shows that the adsorption process depends significantly on the type of activating agent used in the preparation of activated carbon.



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Evaluation of coagulating efficiency and water borne pathogens reduction capacity of *Moringa oleifera* seed powder for treatment of domestic wastewater from Zomba, Malawi

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ABSTRACT

For many communities in the developing world, conventional water treatment methods are often unaffordable because of the high cost associated with them and inavailability of chemical coagulants in the developing countries. Employing *Moringa oleifera* seed (as powder or extracts) to treat municipal domestic wastewater effluent presents an alternative practice to improving water quality effluent of existing wastewater treatment plants in developing countries. In the present study, domestic wastewater from a local wastewater treatment plant in Zomba, Malawi, was treated by *Moringa oleifera* seed powder in batch tests. The objective was to investigate the potential of *Moringa oleifera* seed powder in enhancing domestic wastewater treatment through the reduction of microbial load, turbidity and total dissolved solids (TDS). *Moringa oleifera* powder seed reduced turbidity from 287 to 38.8 Nephelometric turbidity unit (NTU), increased pH from 4.3 to 7.1, and set total dissolved solids (TDS) at standards recommended by World Health Organization guidelines for drinking water. Optimum reduction in microbial load was observed at a dosage of powder of 15 g L^{-1}, with particular potency against *Salmonella* and *Shigella* spp. However, each dose of *Moringa oleifera* seed powder showed its own ideal settling (contact) time for microbe reduction before regrowth of microbes.

Example of NaCl plant extract used in wastewater treatment

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IMPROVEMENT OF THE FLOCCULATION PROCESS IN WATER TREATMENT BY USING *Moringa oleifera* SEEDS EXTRACT

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River water, taken from the Guadiana river at Badajoz (Spain)
(Characterization of raw wastewater)

Parameter	Value	Units
Conductivity	400	$\mu S\ cm^{-1}$
pH	7.5	
Suspended solids	15	$mg\ L^{-1}$
Total solids	452	$mg\ L^{-1}$
Turbidity	123.3	NTU
Calcium	37.7	$Ca^{2+}\ mg\ L^{-1}$
Hardness	152	$CaCO_3\ mg\ L^{-1}$
Ammonium	1.81	$N\ mg\ L^{-1}$
Nitrate	5.3	$NO_3^-\ mg\ L^{-1}$
Nitrite	0.033	$N\ mg\ L^{-1}$
Chloride	40.4	$Cl^-\ mg\ L^{-1}$
$KMnO_4$ oxidizability	34.6	$O_2\ mg\ L^{-1}$
Phosphate	0.044	$P\ mg\ L^{-1}$
Total phosphorus	0.064	$P\ mg\ L^{-1}$
Total coliforms	800	Colonies per 100 mL
Fecal coliforms	400	Colonies per 100 mL
Fecal streptococcus	140	Colonies per 100 mL

Optimization steps

- **pH**: Variation of pH between 3.0 – 9.0
- **Stirring Time**: Variation of the agitation time in the slow stages between 5 and 60 minutes
- **Coagulant dosage**: Variarion of dosage: 8 and 16 mg L^{-1}

Turbidity removal = 98 %

96 % and 94 % removal in the case of total and fecal coliforms

100 % removal in the case of fecal streptococcus

Example of Microfiltration used in wastewater treatment

Coagulation/Flocculation with *Moringa oleifera* and Membrane Filtration for Dairy Wastewater Treatment

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Initial characteristics of the DW, residual values of physicochemical parameters after CFS treatment with 3000 mg/L of coagulant, and removal efficiency of the CFS process

Parameter	Initial values	Residual values	Removal efficiency (%)
COD (mg O_2/L)	4610	4103	11
Color (mg Pt-Co/L)	4141	1925	53
Particle size	7560.5	1907.2	–
pH[a]	4.03	4.6	–
PDI	0.791	0.536	–
Turbidity (NTU)	520	208	60
Zeta potential	− 8.45	0.42	–

[a] Only residual value

The application of CFD process enhanced the microfiltration process

A plant based coagulante was used

There was a high removal of turbidity from the dairy wastewater

To our knowledge, the combined coagulation-flocculation-decantation-microfiltration processes were never applied to winery wastewater treatment and its effects in organic carbon, turbidity, total sus-pended solids and phenolic compounds reduction are unknown.

Therefore, the objectives of this work are


(1) to characterize the plants


(2) to evaluate the production and application of NaCl plant extracts in CFD process


(3) to evaluate the effect of microfiltration in WW treatment


(4) to study the effect of CFD process in microfiltration enhancement and cost reduction

Winery wastewater characterization

Parameters	Portuguese Law Decree nº 236/98	WW
pH	6.0-9.0	4.0±0100
Electrical conductivity (µS/cm)		62.5±0.361
Turbidity (NTU)		296±2.000
Total suspended solids – TSS (mg/L)	60	750±1.528
Chemical Oxygen Demand - COD (mg O_2/L)	150	2145±1.000
Biochemical Oxygen Demand - BOD_5 (mg O_2/L)	40	550±1.155
Total Organic Carbon – TOC (mg C/L)		400±4.040
Total Nitrogen – TN (mg N/L)	15	9.07±0.010
Total polyphenols (mg gallic acid/L)	0.5	22.6±0.100
Biodegradability – BOD_5/COD		0.26±0.015
Aluminium (mg/L)	10.0	0.00±0.000
Calcium (mg/L)		1.07±0.010
Cobalt (mg/L)		0.00±0.000
Copper (mg/L)	1.0	0.014±0.001
Iron (mg/L)	2.0	0.05±0.006
Magnesium (mg/L)		0.51±0.006
Manganese (mg/L)	2.0	0.016±0.001
Potassium (mg/L)		20.5±0.015
Sodium (mg/L)		0.19±0.007
Zinc (mg/L)		10.53±0.006

Production of wastewater

Production of wine

Collection

Storage at -40ºC

Solid part preparation

- Separation of pollen, seeds and rachis
- Washing and drying at 70ºC/ 24h
- Crushing
- Sieve
- Drying at 70ºC/ 24 h

Laboratory incubator

Groundnut miller

Sieve
(Mesh of 150 µm)

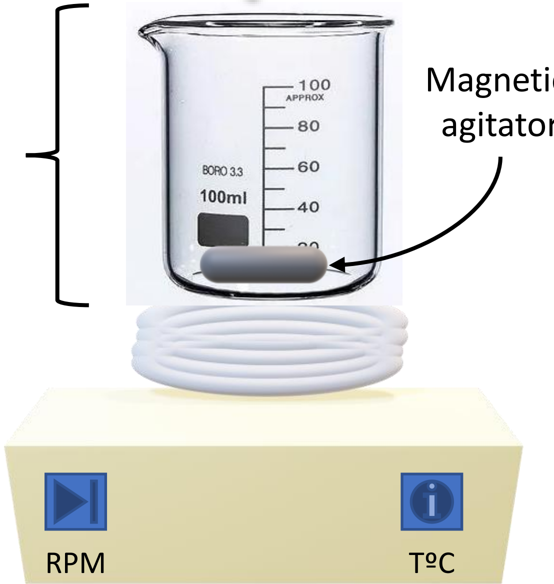
NaCl-plant extract preparation

Plant powder

NaCl 1 M

Gobelet

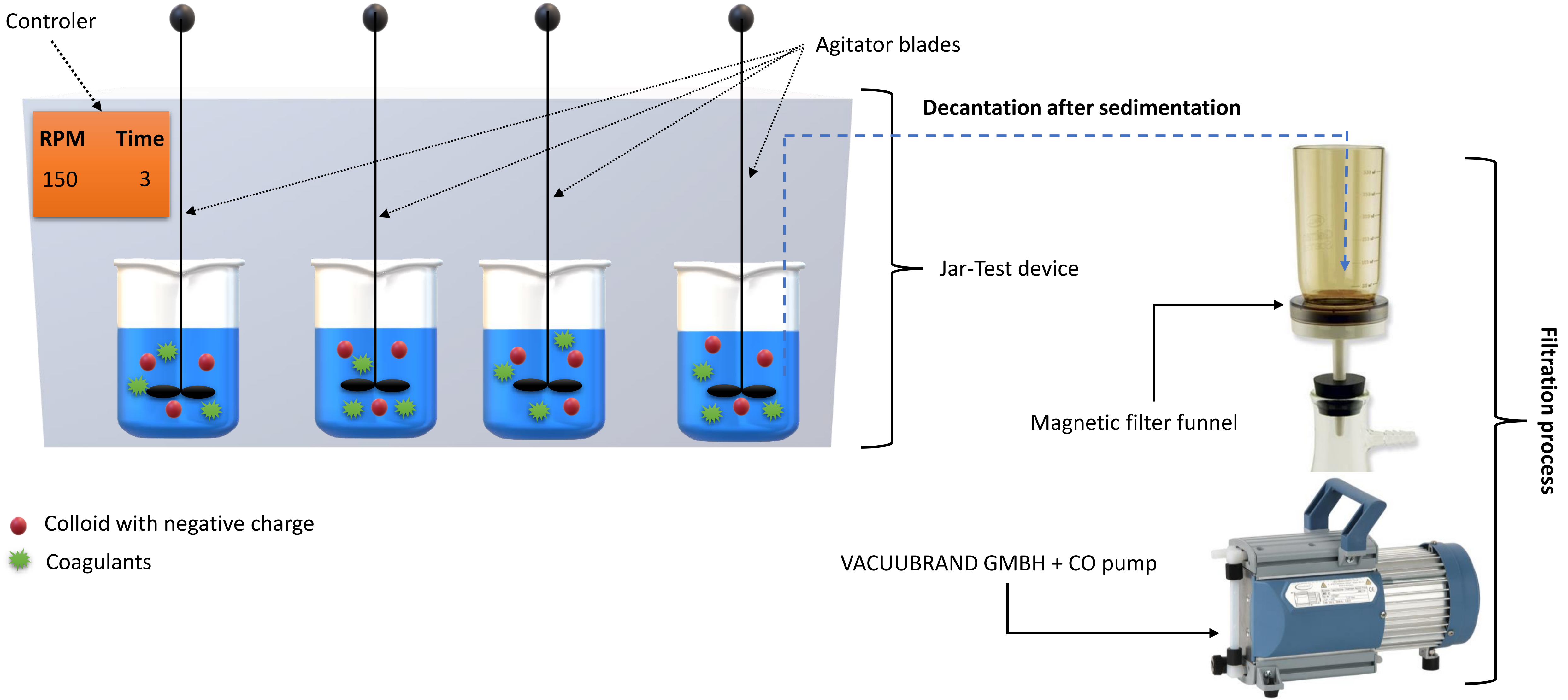
Magnetic agitator



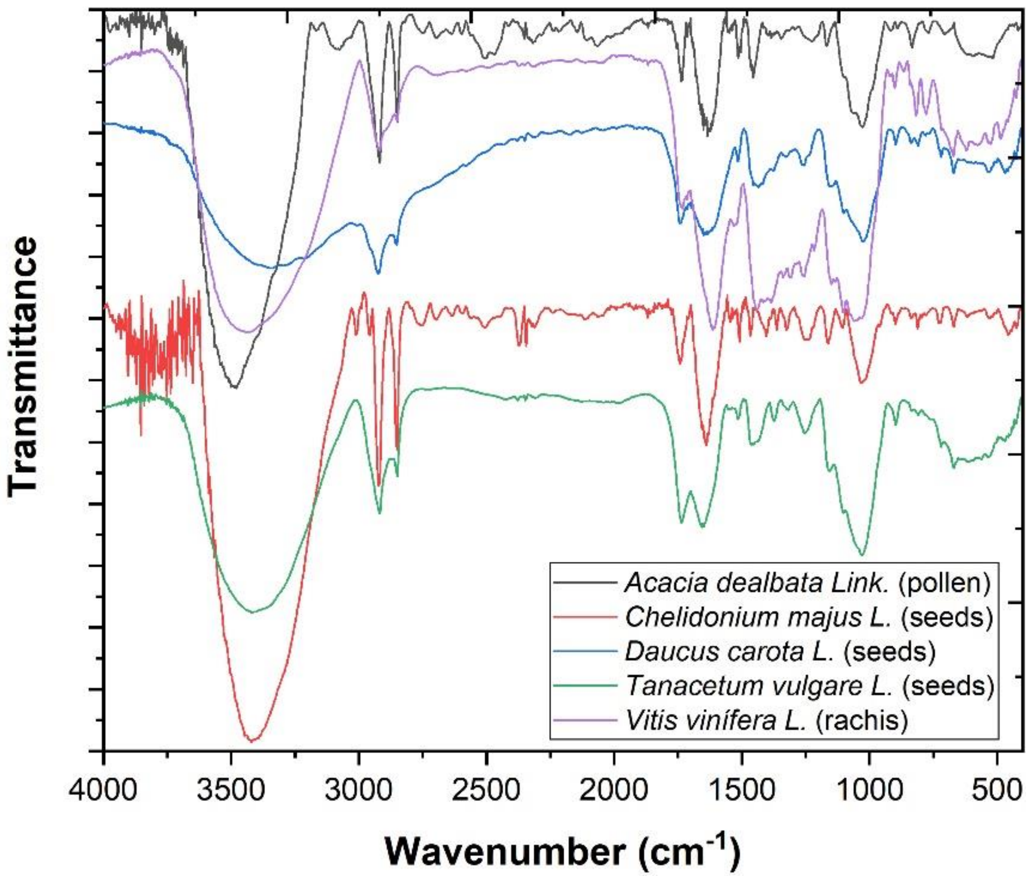
Magnetic agitator plate

- Mixture for 24 h, for extraction of compounds;
- Decantation and filtration of extract

CFD/Microfiltration Setup



Characterization of plants

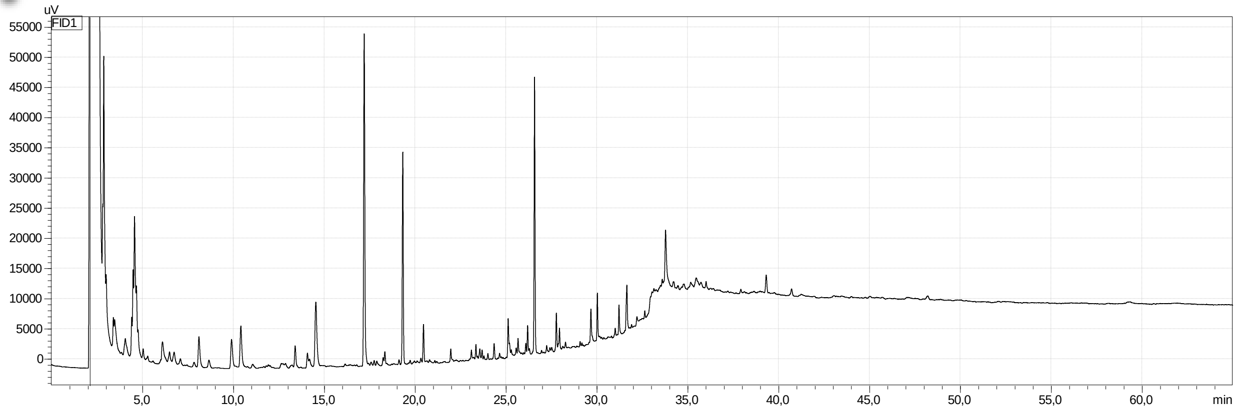


FTIR spectrum of *Acacia dealbata Link*. (pollen), *Chelidonium majus L*. (seeds), *Daucus carota L*. (seeds), *Tanacetum vulgare L*. (seeds) and *Vitis vinifera L*. (rachis).

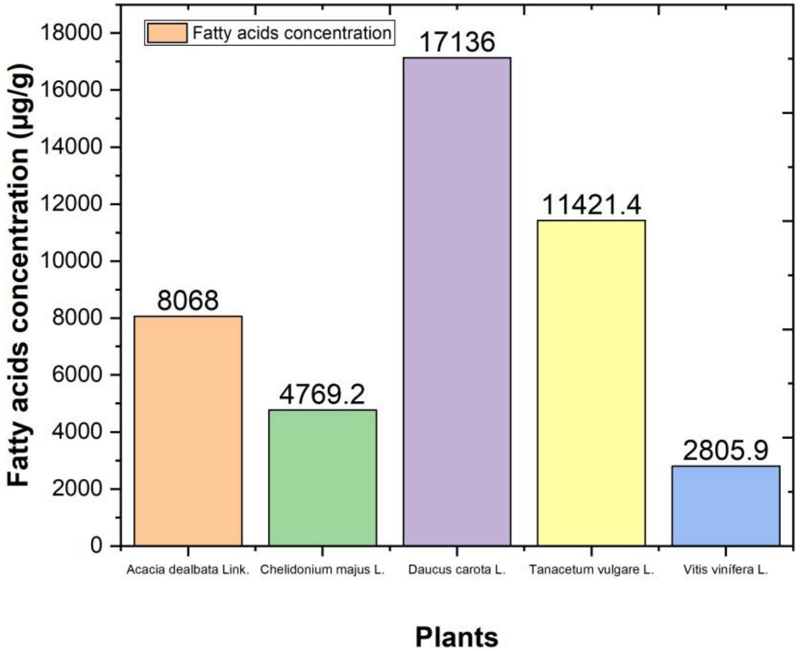
Wavenumber (cm^{-1})	Compound
3421.72	stretching vibrations of OH groups (from water, alcohols, phenols, carbohydrates, per-oxides)
2920.23 and 2850.79	C-H stretching vibrations specific to CH_3 and CH_2 from lipids, metoxy derivatives, C-H (aldehydes), including cis double bonds
1741.72	C-O stretching vibrations of glycerides
1639.49	bending vibrations N-H (amino acids), C=O stretchings (aldehydes and cetones, esters) and free fatty acids
1028.06	C–O stretching vibration from the glucose ring vibration and the holocellulose and hemicellulose
1200 – 1000	C–O–C symmetrically stretching vibration and the aromatic C–H in-plane bending vibrations

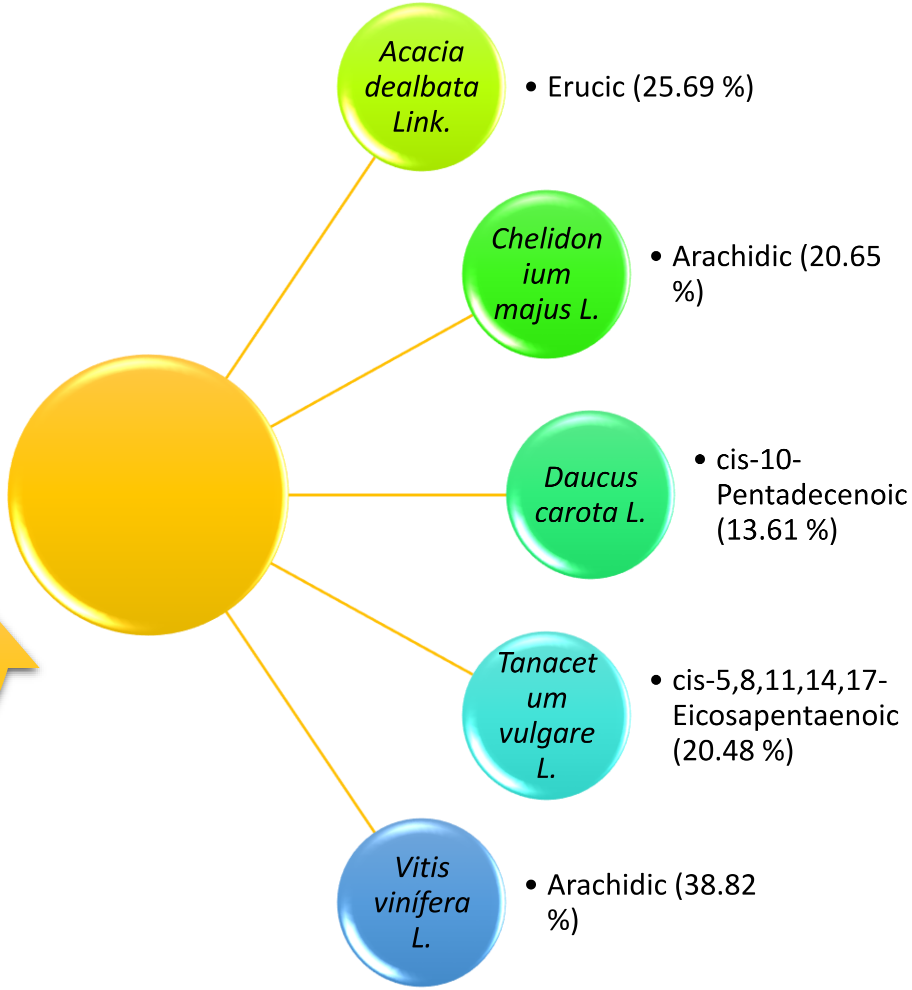
Characterization of plants

- 37 FAME were initialy analysed by **GC-FID** and peaks were compared with plants;
- Results showed diferences between fatty acid composition in each plant.



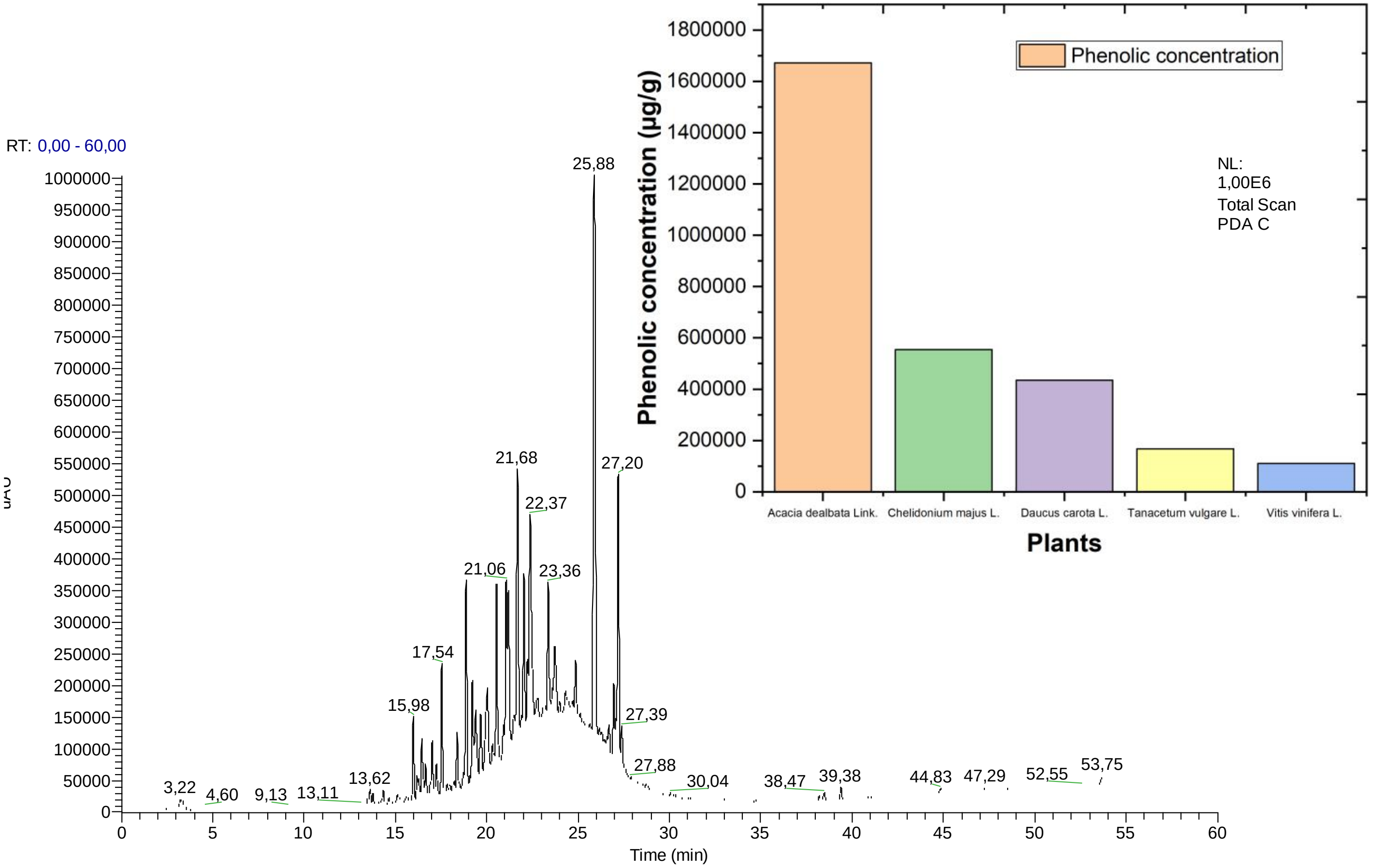
Fatty acid determination by **GC-FID**



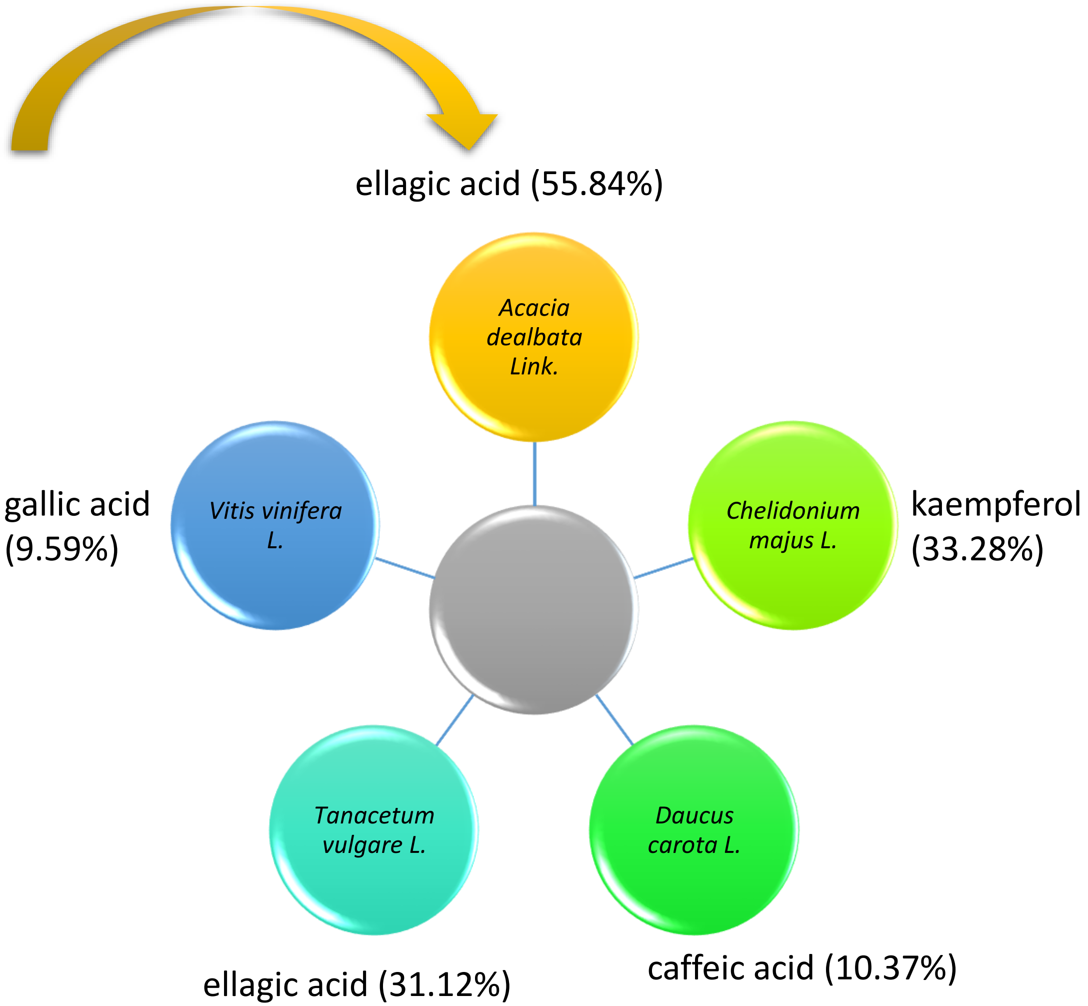


Characterization of plants

- 34 phenolic compounds were initialy analysed by **HPLC-DAD** and peaks were compared with plants;
- It were separated 4 major types of phenolic compounds (hydroxybenzoic acids (280 nm), cinnamic acids (320 nm), flavonoids (350 nm) and anthocyanins (520 nm));
- Results showed diferences between the phenolic composition in each plant.



Phenolic compounds determination by **HPLC-DAD**



Coagulation-flocculation-decantation-microfiltration

Best operational conditions of NaCl plant extract *Acacia dealbata Link*. (pollen), *Chelidonium majus L.* (seeds), *Daucus carota L*. (seeds), *Tanacetum vulgare L*. (seeds) and *Vitis vinífera L*. (rachis) and aluminium sulfate for CFD process, as follows: $[TOC]_0$ = 400 mg C/L, turbidity = 296 NTU, TSS = 750 mg/L, temperature 298 K, sedimentation time 12 h, pump flow rate of 1.9 m^3/h, glass microfiber filters, with micrometric retention of 1.2 µm.

	Phase one						
	Phase two						
	Phase three						
	Phase four						
Coagulant	pH	Dosage	Fast mix	Slow mix	Flocculant type	Flocculant dosage	CFD → Microfiltration
		g/L	rpm/min	rpm/min		mg/L	
Acacia dealbata Link. (5%)	3	0.5	150/3	20/20	Activated sodium bentonite	50	Glass microfiber filters
Chelidonium majus L. (5%)	3	0.5	150/2	50/30	Activated sodium bentonite	5	
Daucus carota L. (5%)	3	0.5	150/3	20/20	Activated charcoal	5	
Tanacetum vulgare L. (5%)	3	0.1	150/3	20/20	Potassium caseinate	100	
Vitis vinífera L. (5%)	3	0.1	150/3	20/20	Potassium caseinate	5	
Aluminium sulfate (10%)	5	0.5	150/2	50/30	Activated charcoal	50	

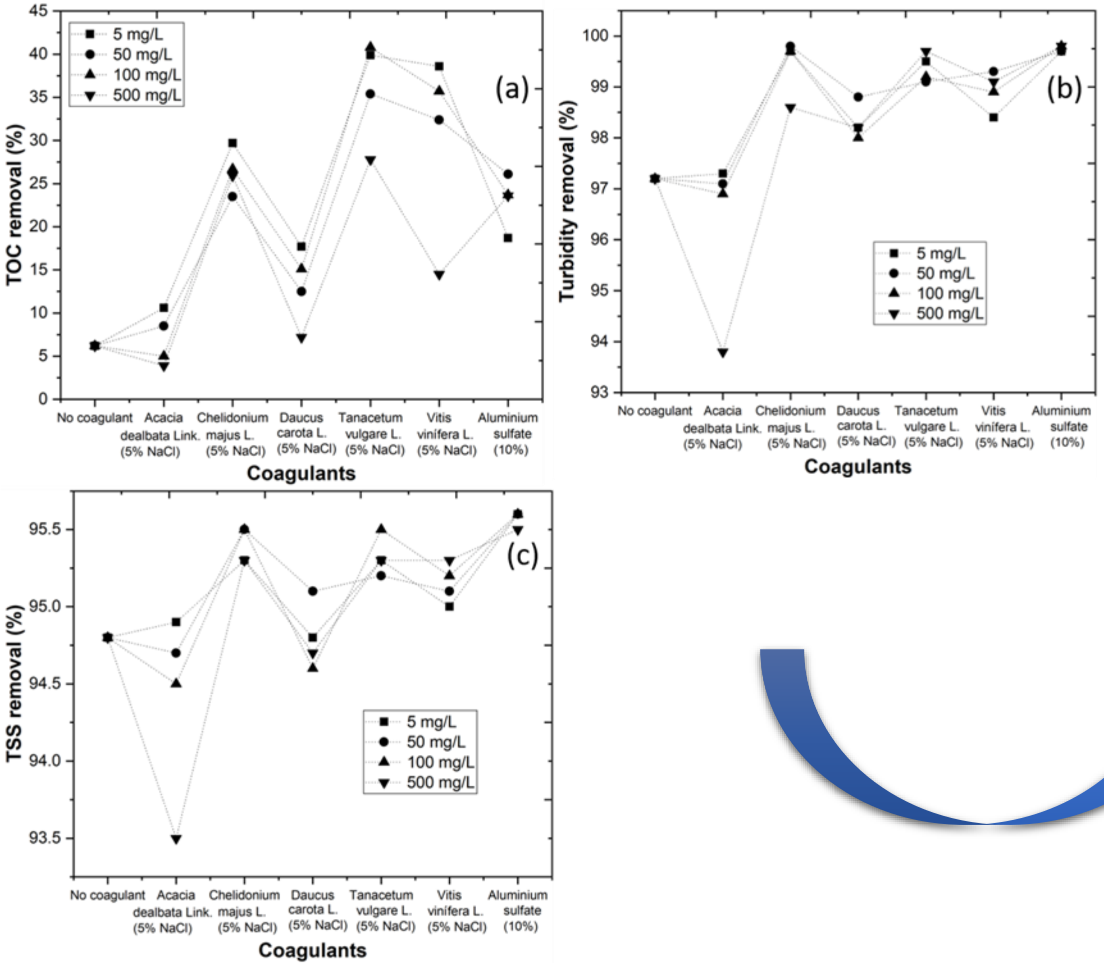
Phase one showed that NaCl plant extracts are more efficient at pH 3.0

The NaCl plant extracts require similar or lower dosage than aluminium sulfate

Some flocculants are more efficient when combined with the coagulants

The CFD process enhanced the microfiltration efficiency

Coagulation-flocculation-decantation-microfiltration



Evolution of (a) TOC, (b) turbidity and (c) TSS removal at different flocculant concentrations (5 – 500 mg/L) in combined coagulation-flocculation-decantation-microfiltration process. CFD/MF operational conditions, as follows: $[TOC]_0$ = 400 mg C/L, turbidity = 296 NTU, TSS = 750 mg/L, temperature 298 K, sedimentation time 12 h, pump flow rate of 1.9 m^3/h, glass microfiber filters, with micrometric retention of 1.2 µm.

Results showed that performance of microfiltration in raw WW and in coagulated WW with NaCl plant extract *Acacia dealbata Link*. (pollen), *Chelidonium majus L*. (seeds), *Daucus carota L*. (seeds), *Tanacetum vulgare L*. (seeds) and *Vitis vinífera L*. (rachis) and aluminium sulfate had a :

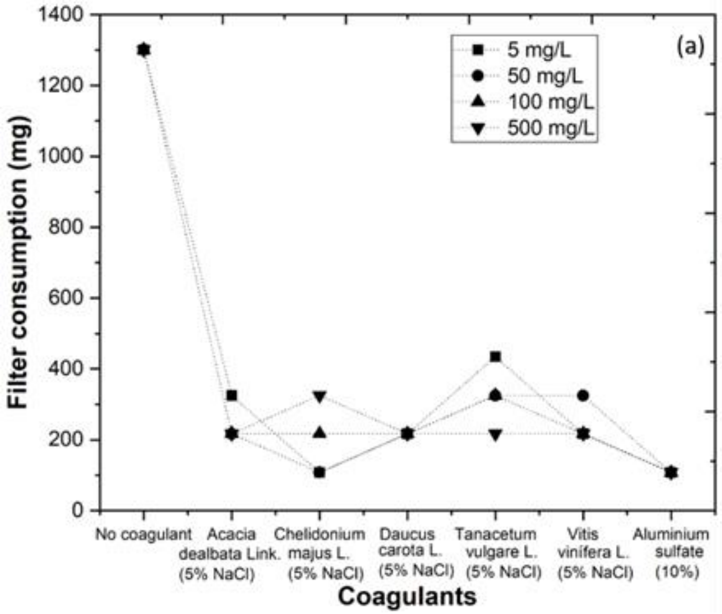
TOC removal of 8.4, 8.5, 29.7, 17.7, 40.8, 38.6 and 26.1%

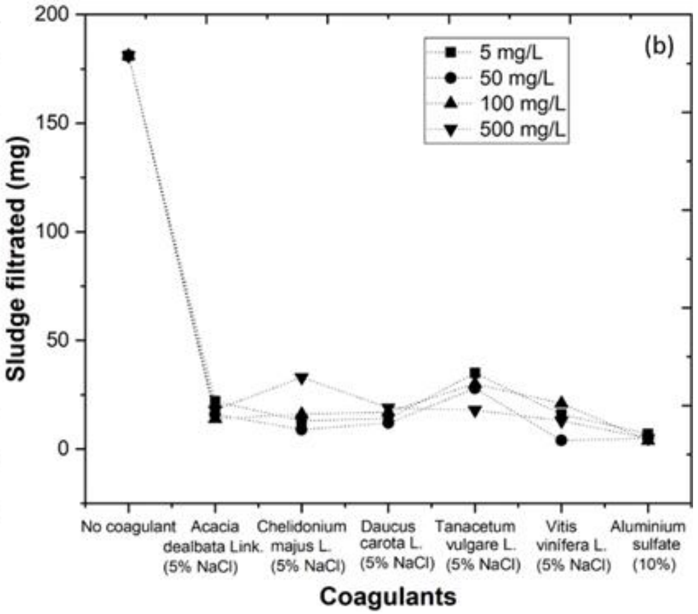
Turbidity removal of 97.2, 97.1, 99.7, 98.2, 99.2, 98.4 and 99.7%

TSS removal of 94.8, 94.7, 95.3, 94.8, 95.5, 95.0 and 95.6%

Coagulation-flocculation-decantation-microfiltration

Results showed that performance of microfiltration in raw WW and in coagulated WW with NaCl plant extract *Acacia dealbata Link*. (pollen), *Chelidonium majus L*. (seeds), *Daucus carota L*. (seeds), *Tanacetum vulgare L*. (seeds) and *Vitis vinífera L*. (rachis) and aluminium sulfate had a :





Filter consumption of 1301, 217, 108, 217, 325, 217 and 108 mg

Higher costs derived mainly from the presence of sediments in the WW (181, 16, 13, 14, 30, 16 and 5 mg, respectively)

Positive correlation between **sludge compaction** by coagulation-flocculation-decantation process, and **filter consumption** by microfiltration process ($y = 2.55x + 12.68$, $r^2 = 0.949$), which indicated that CFD process had a direct effect in microfiltration cost reduction

CFD/MF operational conditions, as follows: $[TOC]_0$ = 400 mg C/L, turbidity = 296 NTU, TSS = 750 mg/L, temperature 298 K, sedimentation time 12 h, pump flow rate of 1.9 m^3/h, glass microfiber filters, with micrometric retention of 1.2 µm.

With the results from this work, it is concluded that

(1) It is possible to produce NaCl-plant extracts and apply them as coagulants

(2) The Na-Cl plant extracts enhance the microfiltration process and decrease the costs

(3) The application of CFD/MF process is an economic and sustainable technology for WW treatment

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