Effects of cleaning procedures on the concentration of pesticide residues on crisp fresh-cut lettuce (cv. Vera)

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Introduction

Consumption of ready-to-eat vegetables has increased due to new trends in diet and lifestyle, thus having an impact on both commercial and domestic environments. Pesticide usage during large-scale production of fruits and vegetables can be problematic as it´s presence, though useful for preventing plagues and diseases, should be closely monitored along the whole process to ensure the safety of the product. Each part of the process needs to be taken into consideration, particularly the post-harvest stages[1,2]. Non-thermic treatments, like ultrasound baths, are being used as an alternative for disinfection which have the potential to lower pesticide residues. Ultrasound in particular is a safe, non-toxic and clean technology that allows for scaling and continuous design, therefore it is already being used at industrial level.[3] On the other hand, there are "domestic" types of cleaning procedures, considered safe and non-toxic, that are not to be forgotten, and also can be attached to industrial scale processes.

Objective

The aim of this work was to test and compare the effectiveness of different cleaning procedures to reduce pesticide residues on crisp fresh-cut lettuce.

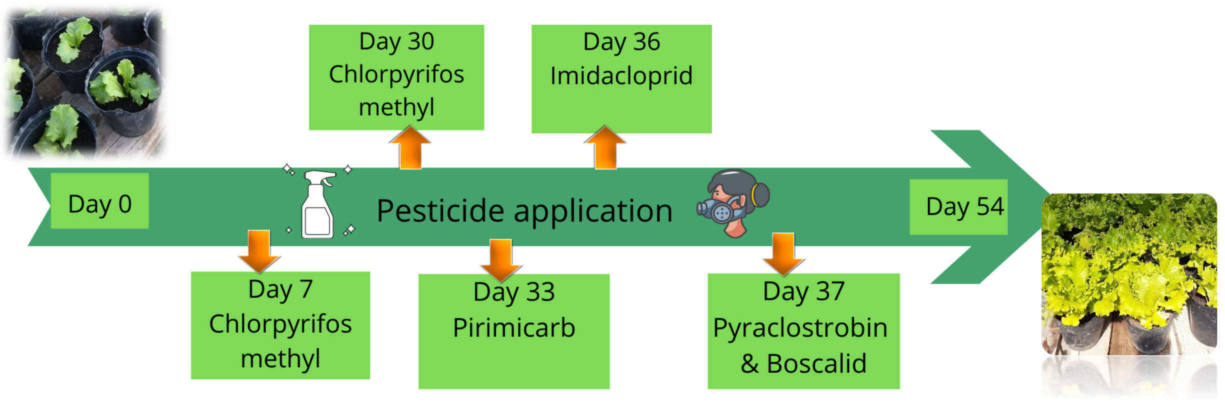
Methodology

Field experiment

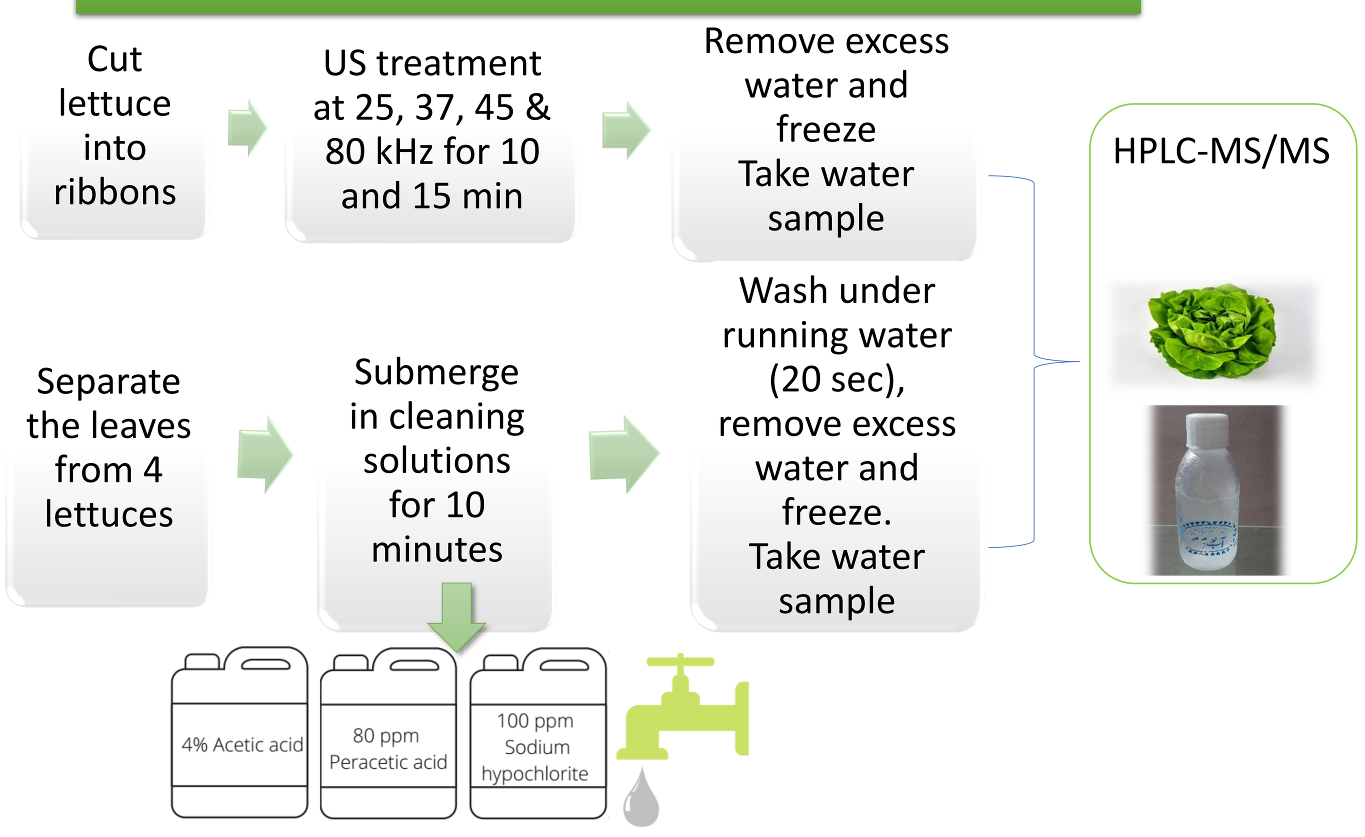
Incurred samples: Two groups of crisp lettuce were cultivated under controlled conditions for 54 days in a greenhouse with daily watering. Group A consisted of 6 lettuces while group B consisted of 64 plants.

Pesticide application:
Group A: without pesticide application (**control**).
Group B: Pesticide application according to Good Agricultural Practices.[4]

Selected Pesticides: Chlorpyrifos methyl, Pirimicarb, Imidacloprid, Boscalid, Pyraclostrobin.



Treatments



Comercial samples

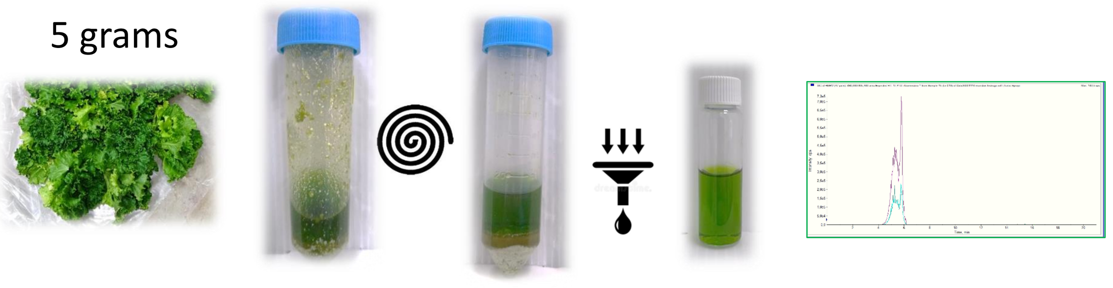
22 commercial lettuces were acquired from different shops in Paysandú to evaluate pesticide residues level by HPLC-MS/MS.

Sample processing

The samples were cut in pieces and then milled with liquid nitrogen and kept frozen until analysis

Pesticide extraction (water samples)[5]

Pesticide extraction (acetate QuEChERS)



Results and Discussion

A QuEChERS method was developed and validated for the analysis of 17 pesticides widely used in lettuce crops

Pesticide	10 µg/kg		25 µg/kg		50 µg/kg	
	RSD	%Rec	RSD	%Rec	RSD	%Rec
Abamectin	7	120	13	85	12	101
Acetamiprid	1	87	3	96	2	98
Boscalid	9	91	4	100	4	99
Carbendazim	1	59	3	82	3	102
Chlorpyrifos	2	99	4	106	4	108
Cyromazine	18	94	3	85	3	80
Chlorpyrifos methyl	2	84	7	109	3	85
Dimethoate	1	97	2	96	3	91
Fluvalinate	7	68	14	95	12	124
Iprodione	---	---	---	---	7	91
Lambda cyhalothrin	12	87	13	116	10	114
Methomyl	1	102	4	99	3	90
Pirimicarb	2	77	4	94	3	94
Propamocarb	2	83	4	88	2	96
Pyraclostrobin	5	71	1	93	2	97
Pyrimethanil	4	76	4	88	6	99
Spinosad	2	101	4	99	3	97

Table 1: Standard deviation and recovery percentages for all pesticides.

12 pesticides LOQ= 5 µg/kg for 12 pesticides
LOQ= 10 µg/kg, Pirimicarb, Lambda cyhalothrin, and Boscalid
LOQ = 25 µg/kg Abamectin

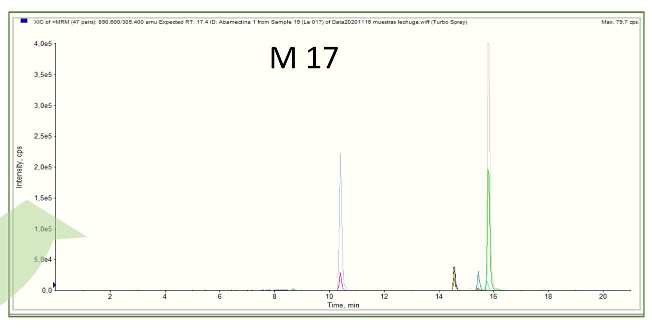
Most pesticides presented a quantitation limit of 10 µg/kg, except for Carbendazim 25 µg/kg and Iprodione 50 µg/kg.

Matrix effect was low (<20%) for most pesticides, except for carbendazim, therefore this method can be used without the need of a matrix matched calibration curve.

Pesticide	MRL (mg/kg)	M 12 (mg/kg)	M 13 (mg/kg)	M 14 (mg/kg)	M 16 (mg/kg)	M 17 (mg/kg)	M 18 (mg/kg)
Acetamiprid	---	ND	ND	ND	0.12	0.41	ND
Boscalid	---	ND	ND	ND	0.32	0.99	ND
Iprodione	10	ND	ND	ND	0.05	0.13	ND
Propamocarb	100	2.5	4.2	0.03	ND	ND	10
Pyraclostrobin	40	ND	ND	ND	0.125	0.31	ND

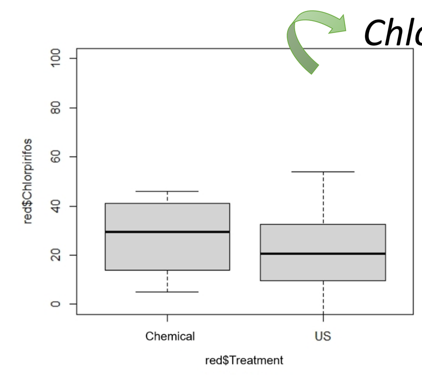
Table 2: Results of the positive commercial samples.

Total ion chromatogram obtained through HPLC-MS/MS from a sample.



Six out of the 22 commercial samples presented pesticides residues over the LOQ, but all of them were under the MRLs according to de *Codex Alimentarius*[6]

Chlorpyrifos methyl box/plot diagram for domestic and ultrasound treatments.



ANAVA test Pillai-Bartlett with alfa 0,05.
The obtained p/value was 0.28, therefore, no significant differences were found.

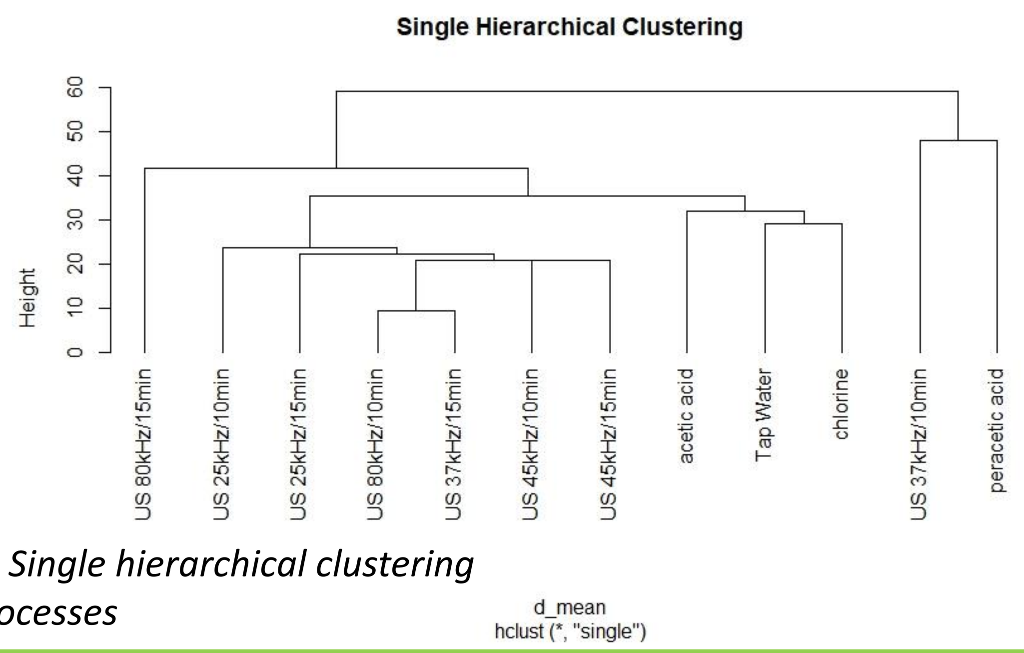
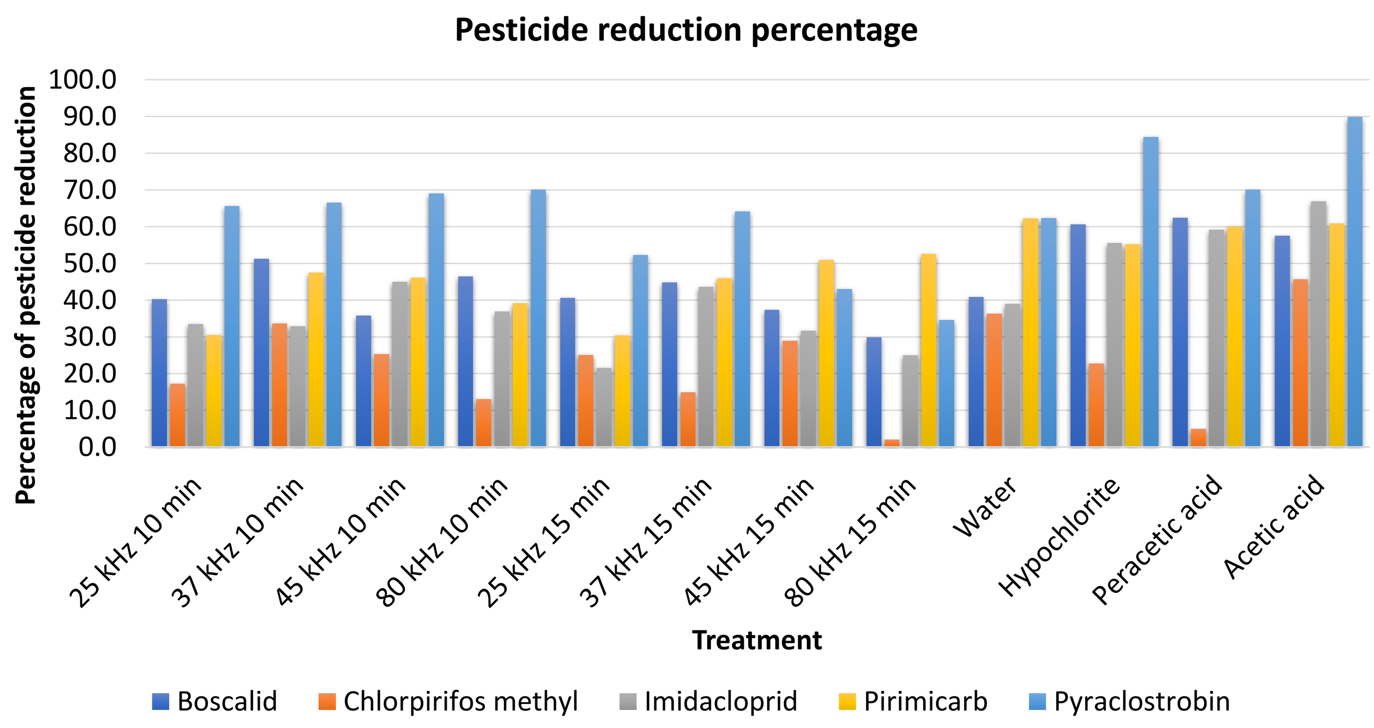


Image 1: Single hierarchical clustering for all processes



Graphic 1: Pesticide residue reduction for each treatment

Conclusions and future work

- A fit for purpose methodology for the determination of pesticides residues in lettuce was developed and validated.
- The applicability of the method was tested in the analysis of 22 commercial samples, where seven showed pesticide residues
- No differences were found between the ultrasound treatments at the selected frequencies and times nor between them and the domestic treatments.
- All treatments managed to reduce pesticide residues, with chlorpyrifos methyl being the most resilient.
- No pesticide residues were found at concentrations higher than the LOQ in the water samples.

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