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Toward efficient recovery of the alkaloid plantagonine from *Verbascum sinuatum* flowers: Optimization of microwave-assisted and pressurized liquid extraction methods

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1 INTRODUCTION

The increasing scientific interest in ***Verbascum sinuatum* flowers** (**VSFs**) is due to their potential applications across multiple sectors, including the pharmaceutical, cosmeceutical, and food industries, particularly in the development of functional foods and nutraceuticals. The phytochemical composition of VSFs is predominantly characterized by phenolic compounds, iridoid glycosides, and alkaloids such as plantagonine. This bioactive alkaloid identified in VSFs has been reported to have several biological activities such as antioxidant, anti-inflammatory and immunomodulating activities amongst others. In the literature, no studies have addressed the optimization of plantagonine extraction from VSFs. Thus, this study aimed to **maximize the extraction of plantagonine** from VSFs through microwave-assisted extraction (**MAE**) and pressurized liquid extraction (**PLE**) using the response surface methodology (**RSM**) and five level Circumscribe central composite design (**CCCD**).

2 MATERIAL AND METHODS

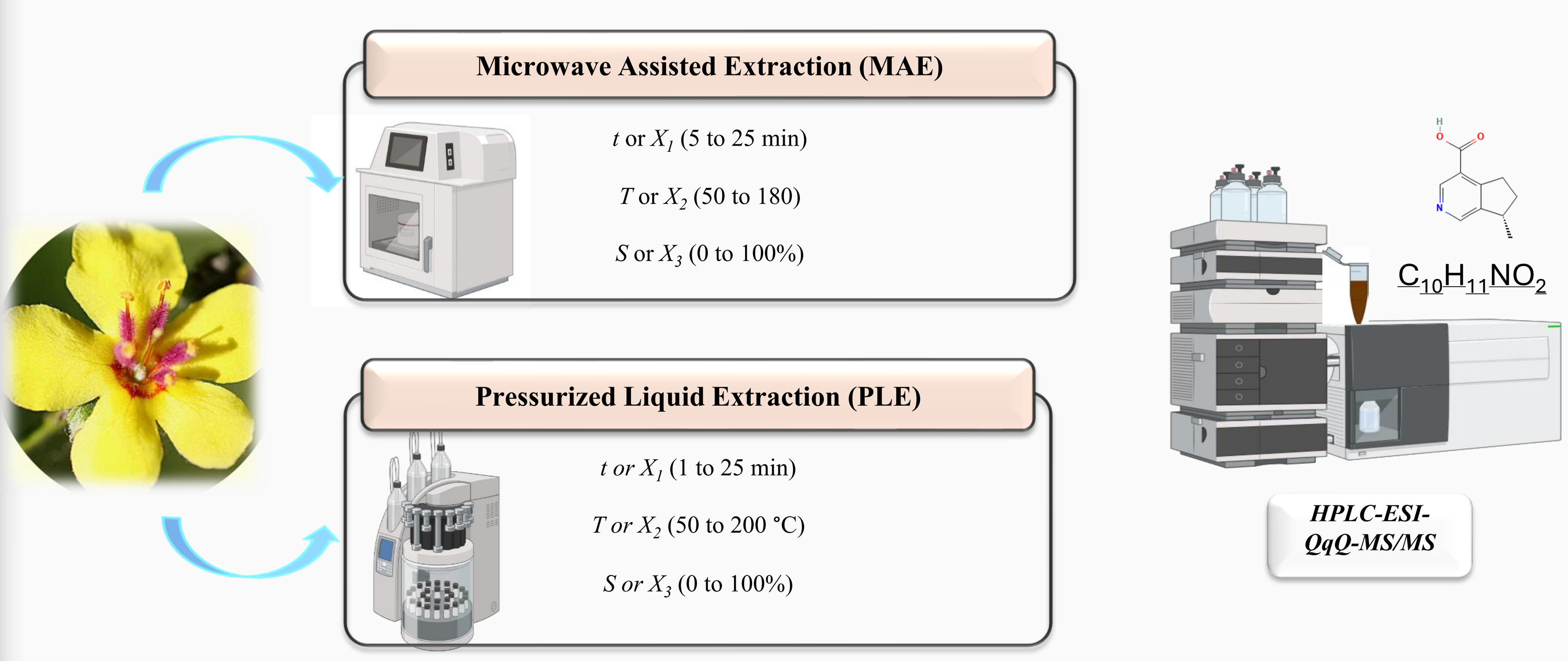


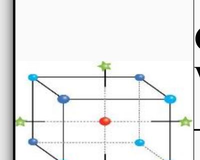
Table 1. Experimental plan and experimental results

CODIFIED VALUES				*Plantagonine (μg/g E)*	
Runs	X_1	X_2	X_3	*MAE*	*PLE*
1	-1	-1	-1	427.64	898.26
2	-1	-1	1	666.36	651.31
3	-1	1	-1	413.04	1226.91
4	-1	1	1	892.05	823.76
5	1	-1	-1	513.78	717.53
6	1	-1	1	541.51	608.31
7	1	1	-1	831.10	1001.30
8	1	1	1	802.20	1011.47
9	1.68	0	0	1128.24	1766.47
10	-1.68	0	0	978.57	1391.21
11	0	-1.68	0	932.17	657.31
12	0	1.68	0	1003.67	2144.02
13	0	0	-1.68	189.86	705.24
14	0	0	1.68	408.76	146.16
15	-1.68	-1.68	-1.68	288.16	154.25
16	-1.68	-1.68	1.68	149.98	183.00
17	-1.68	1.68	-1.68	633.63	1238.76
18	-1.68	1.68	1.68	413.24	580.85
19	1.68	-1.68	-1.68	138.27	254.53
20	1.68	-1.68	1.68	187.10	27.03
21	1.68	1.68	-1.68	699.57	1969.49
22	1.68	1.68	1.68	382.74	1767.16
23	0	0	0	682.27	1744.65
24	0	0	0	658.74	1579.17
25	0	0	0	1011.20	1789.02
26	0	0	0	712.15	1546.91
27	0	0	0	670.16	1470.82
28	0	0	0	877.59	1363.51

Data fitting through least square method

$$Y = b_0 + \sum_{i=1}^{n} b_i X_i + \sum_{\substack{i=1 \\ j>i}}^{n-1} \sum_{j=2}^{n} b_{ij} X_i X_j + \sum_{i=1}^{n} b_{ii} X_i^2 + \sum_{\substack{i=1 \\ j>i}}^{n-1} \sum_{j=2}^{n} b_{iijj} X_i^2 X_j^2 + \sum_{\substack{i=1 \\ j>i}}^{n-2} \sum_{j=2}^{n-1} \sum_{\substack{k=3 \\ k>j}}^{n} b_{ijk} X_i^2 X_j^2 X_k^2 + \sum_{i=1}^{n} b_{iii} X_i^3$$

Table 2. Experimental variables tested, the intervals and the codes for calculating the CCCD distribution with five levels of values.



CODIFIED VALUES	DECODIFIED VALUES					
	MAE			PLE		
	t (min)	*T* (ºC)	*S* (%)	*t* (min)	*T* (ºC)	*S* (%)
-1.682	5	50	0	1	50	0
-1	9.1	76.4	20.3	5.9	80.4	20.3
0	15	115	50	13	125	50
1	20.9	153.6	79.7	20.1	169.6	79.7
1.682	25	180	100	25	200	100

3 RESULTS AND DISCUSSION

Table 3. Parametric information, coefficient of determination and optimal extraction condition of plantagonine from VSFs through MAE and PLE

		Coefficient	MAE		PLE	
1	Intercepción	b_0	767.469	±67.954	1534.01	±90.04
2	Time	X_1	128.687	±92.654	--	--
3	Temperature	X_2	--	--	--	--
4	Solvent	X_3	226.881	±92.654	--	--
5	Time^2	X_1^2	86.711	±44.943	--	--
6	Temperature^2	X_2^2	56.489	±44.943	-108.74	±70.20
7	Solvent^2	X_3^2	-179.900	±44.943	-453.44	±70.20
13	Time^3	X_1^3	-43.977	±36.464	44.30	±17.16
14	Temperature^3	X_2^3	30.892	±10.878	135.51	±17.16
15	Solvent^3	X_3^3	-88.785	±36.464	--	--
8	Time*Temperature	X_1*X_2	--	--	80.08	±30.96
9	Time*Solvent	X_1*X_3	--	--	--	--
10	Temperature*Solvent	X_2*X_3	-20.435	±19.621	--	--
11	Time*Temperature*Solvent	$X_1*X_2*X_3$	--	--	19.37	±19.11
16	Time^2*Temperature^2	$X_1^2*X_2^2$	--	--	--	--
17	Time^2*Solvent^2	$X_1^2*X_3^2$	--	--	--	--
18	Temperature^2*Solvent^2	$X_2^2*X_3^2$	--	--	--	--
12	Time^2*Temperature^2*Solvent^2	$X_1^2*X_2^2*X_3^2$	-13.415	±9.134	36.32	±11.37
		R^2	**0.8598**		**0.9013**	
		t	**25.00**	±2.50	**25.00**	±2.50
		T	**180.00**	±6.71	**200.00**	±7.07
		S	**58.77**	±3.83	**55.00**	±3.71
		Optimum	**1356.20**	±32.10	**2312.86**	±39.69

Extraction yield of the alkaloid plantagonine from VSFs: PLE > MAE

Optimum (MAE&PLE):
- Higher *t*.
- Higher *T*
- Middle %*S*

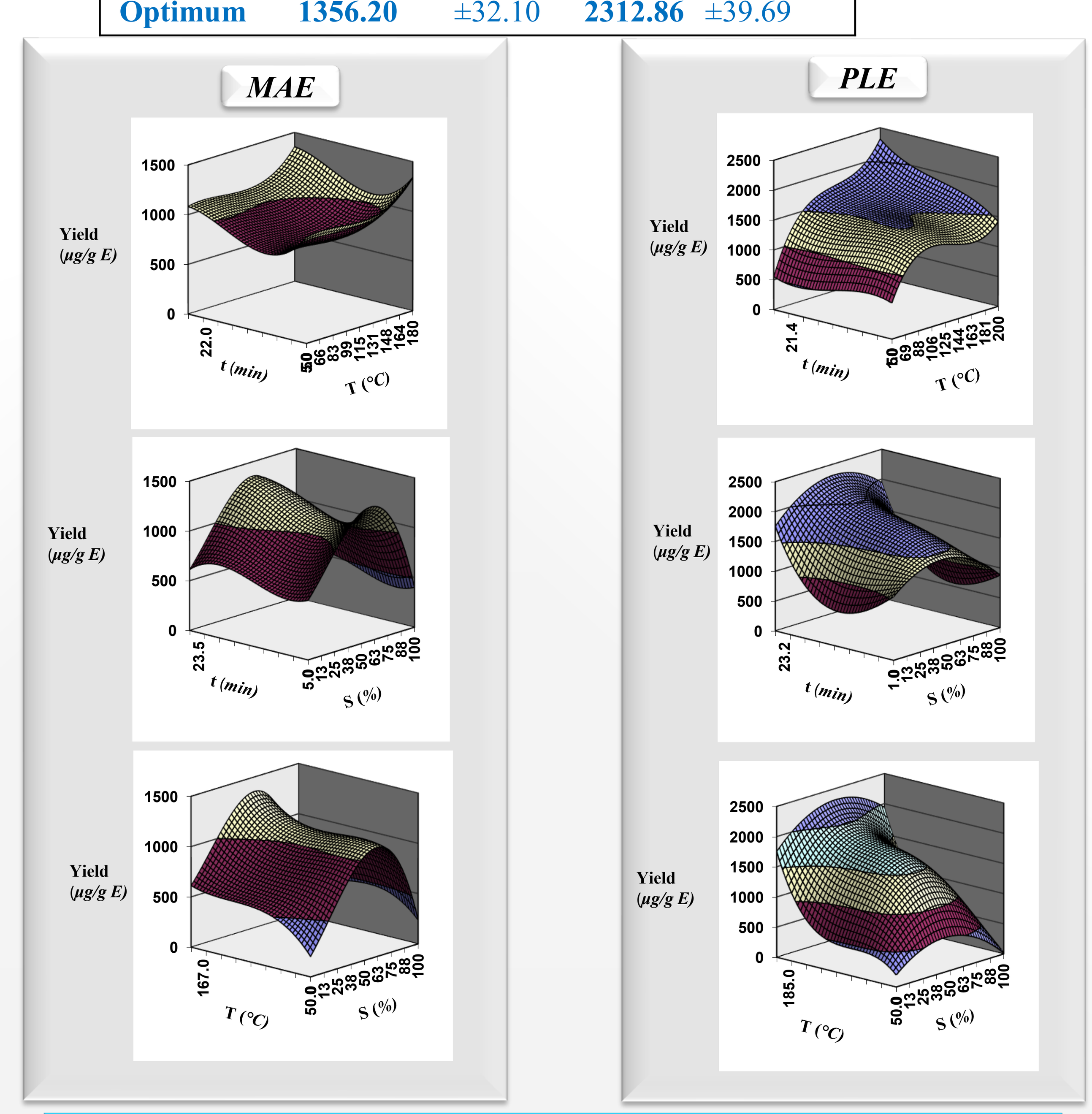


Figure 1. 3D representation of plantagonine recovery from VSFs through MAE and PLE

4 CONCLUSION

Under optimal extraction conditions, PLE reported higher plantagonine recovery yield (2312.86 ± 39.69 µg/g E; 25 min, 200°C, 59% ethanol) with 1.7-fold greater than MAE (1356.20±32.10 µg/g E; 25 min, 180°C, 55% ethanol).

This study proposes, for the first time, the optimal extraction conditions for maximizing the recovery of plantagonine from VSFs using two green extraction techniques, proving the efficiency of PLE, presenting a quantitative framework for further industrial extraction and application in industries in relation to their antioxidant, anti-inflammatory and immunomodulating properties.

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