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Sensitive and Eco-Friendly Determination of Parabens in Toothpaste Using Ionic Liquid-Based DLLME Coupled with HPLC-PDA

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Abstract

An environmentally friendly ionic liquid-based dispersive liquid-liquid microextraction (IL-DLLME) method was developed and validated for the simultaneous determination of four parabens: methylparaben (MP), ethylparaben (EP), propylparaben (PP) and butylparaben (BP) in commercial toothpastes using high-performance liquid chromatography with photodiode array detector (HPLC-PDA). Hydrophobic ionic liquid 1-butyl-3-methylimidazolium hexafluorophosphate [C₄mim][PF₆] and acetonitrile were chosen as extractant and dispersion solvent, respectively. The parameters affecting the extraction efficiency such as pH of the sample solution, dispersion time, volume of the ionic liquid, volume of the dispersion solvent, ionic strength and centrifugation time were optimized by the univariate method. Calibration curves were linear in the range of 100–1000 µg L⁻¹ under optimum conditions with correlation coefficients (R²) of 0.9949–0.9988. The Limits of detection and quantification ranged from 0.69 to 1.01 µg L⁻¹ and 1.23 to 1.70 µg L⁻¹, respectively. Relative standard deviations for intra- and inter-day analyses (n = 5) were less than 8.4%. The method provided enrichment factors of 120–135 and recovery values ranging from 90.4% to 101.5%.

Keywords: Toothpaste, ionic liquid dispersive liquid–liquid microextraction, parabens; HPLC-PDA, preservatives.

1. Introduction

Dispersive liquid-liquid microextraction (DLLME), introduced by Rezaee et al. [1], is one of the most popular miniaturized sample preparation techniques in modern analytical chemistry. The method involves the fast injection of a small volume of water-immiscible extraction solvent, diluted in a miscible disperser solvent, into the aqueous sample to form a fine emulsion of the two phases, which provides an increased interfacial surface area and thus a faster analyte

transfer. Centrifugation is then used to sediment the enriched extraction phase. DLLME provides very short extraction times, high enrichment factors, simple operation, and significantly less organic solvent consumption in comparison with conventional liquid-liquid extraction [1,2].

However, the traditional version of DLLME uses extractants that are dangerous to the human health and environment, such as chlorinated extractants. Therefore, an important step toward more environmentally friendly analytical techniques is the substitution of ionic liquids (ILs) for such solvents. The ionic liquids are molten salts at room temperature that have very low vapor pressure, good thermal stability, and physicochemical characteristics that may be adjusted by combining the right cation and anion [3, 4]. Non-polar ILs with the $[\text{PF}_6]^-$ anion sediment easily in aqueous conditions after centrifugation, which makes them very useful for DLLME. Because of its high density, π - π interactions with aromatic analytes, and moderate hydrophobicity, 1-butyl-3-methylimidazolium hexafluorophosphate $[\text{C}_4\text{mim}][\text{PF}_6]$ has been used extensively as an IL-DLLME extractant [3,5].

Alkyl esters of para-hydroxybenzoic acid, or parabens, are commonly employed as antimicrobial preservatives in food, medicine, cosmetics, and personal hygiene items like toothpaste [6, 7]. The four most often reported parabens are: MP ($\log P = 1.46$), EP ($\log P = 1.80$), PP ($\log P = 2.29$), and BP ($\log P = 2.71$); their variances in alkyl-chain length result in variations in lipophilicity and antibacterial activity [8]. Table 1 summarizes physicochemical characteristics that are important for extraction behavior.

Concerns over long-term paraben exposure have been raised by an increasing amount of toxicological data. Numerous investigations have shown estrogenic activity and possible endocrine-disrupting consequences, including support for hormone-sensitive cancers, including breast cancer [10], and links with reproductive damage [9]. Residual parabens may be absorbed systemically through buccal or gastrointestinal channels after toothpaste comes into direct contact with the mouth mucosa [11]. Sensitive analytical monitoring is crucial because European Cosmetics Regulation No. 1223/2009 does not set any necessary concentration limits for parabens in toothpaste despite the known health risks [12].

Solid-phase extraction (SPE) [13] and cloud-point extraction [14] are two previously documented paraben detection techniques. Toothpaste matrices comprising pulverized silica and surfactants provide special analytical difficulties, as shown by Zotou et al. [11]. When this work was started, no publicly accessible study had ever used IL-DLLME to determine the amount of parabens in toothpaste. Therefore, the goal of the current work is to create, refine, and validate an IL-DLLME-HPLC-PDA method for the simultaneous measurement of MP, EP, PP, and BP in commercial toothpaste products.

2. Related Work

Numerous extraction and detection platforms are used in analytical techniques for paraben measurement. Limits of detection (LOD) of $100\text{--}300\text{ }\mu\text{g L}^{-1}$ were attained by SPE-LC-UV methods applied to saliva and toothpaste [11], which are sufficient for routine screening but insufficient for tracking trace-level contamination. Sensitivity was increased to the $10\text{--}200\text{ }\mu\text{g L}^{-1}$ range by cloud-point extraction in conjunction with HPLC-MS/MS [14] and DLLME in conjunction with capillary electrophoresis [15], however these methods usually ask for large sample quantities or time-consuming tuning. Although it requires nanomaterial manufacturing, activation procedures, and magnetic separation equipment, magnetic solid-phase extraction employing magnetic cellulose nanoparticles (MSPE-MCNPs) [16,8] achieved LODs similar to the current work ($0.69\text{--}1.01\text{ }\mu\text{g L}^{-1}$).

Although IL-DLLME has been used on medicinal chemicals, insecticides, and polycyclic aromatic hydrocarbons [3], its use on parabens in toothpaste has not been reported before our work yet. By utilizing the high density and selective solvation characteristics of imidazolium IL, without the need for chlorinated solvents, the combination of $[\text{C}_4\text{mim}][\text{PF}_6]$

as extractant and MeCN as disperser positions, IL-DLLME, as an environmentally friendly substitute, simultaneously satisfies sensitivity requirements and minimizes laboratory chemical waste [17].

3. Methodology

3.1. Standards, Solvents and Reagents

All reagents were of analytical grade. MP ($\geq 99\%$), EP ($\geq 99\%$), PP ($\geq 98\%$), and BP ($\geq 99\%$) reference standards were obtained from Sigma-Aldrich (St. Louis, MO, USA). The ionic liquid 1-butyl-3-methylimidazolium hexafluorophosphate [C_4mim][PF₆], ($\geq 97\%$) was purchased from Sigma-Aldrich. LC-MS grade acetonitrile (MeCN) and methanol (MeOH) were obtained from Scharlab (Barcelona, Spain). Sodium chloride (NaCl, $\geq 99.5\%$), hydrochloric acid (HCl, 37 % w/w), sodium hydroxide (NaOH, $\geq 98\%$), and phosphoric acid (H₃PO₄, 85 %) were from Sigma-Aldrich. Ultrapure water ($\geq 18.2\text{ M}\Omega\cdot\text{cm}$) was produced by a Milli-Q system (Millipore, Bedford, MA, USA).

3.2. Instrumentation

A Shimadzu Prominence LC-2030C 3D HPLC system (Shimadzu Corporation, Kyoto, Japan) with a photodiode array detector (PDA) was used for chromatographic analysis. At 30°C, separation was accomplished on a Hypersil ODS C18 column (250 mm $\times$ 4.6 mm i.d., 5 μm ; Thermo Scientific, USA). MeOH:water (60:40, v/v) was used as the mobile phase, and it was supplied isocratically at 1.0 mL min⁻¹. The injection volume was 10 μL , and the detection wavelength was 254 nm. Within ten minutes, all four parabens were resolved. Phase separation was carried out using a Hettich Universal 320 centrifuge (Germany). For emulsification, an IKA® MS 3 vortex mixer was utilized. An Orion Star A211 pH meter (Thermo Scientific, USA) was used to measure the sample's pH.

3.3. Preparation of Standard Solutions and Toothpaste Samples

All analyte stock standard solutions were made in MeOH at a concentration of 1 mg L⁻¹ and kept in the dark at 4°C. Every day, mixed working standards were made by serially diluting ultrapure water. Six commercial toothpaste products: standard fluoride toothpaste (FT), whitening toothpaste (WT), charcoal-based toothpaste (CT), children's toothpaste (ChT), herbal toothpaste (HT), and sensitive toothpaste (ST) were bought from pharmacies in Ramallah, Palestine. A mass of 5 mg of each toothpaste was dissolved in a mixture of 3.0 mL MeOH and 7.0 mL water, sonicated for 10 min after addition of 1.0 mL concentrated HCl, then diluted to 150 mL with ultrapure water. The pH was adjusted to the optimum value and the solution was filtered through a 0.45 μm PVDF membrane prior to IL-DLLME.

3.4. IL-DLLME Procedure

A 15 mL glass centrifuge tube with a conical bottom was filled with 10 mL of the aqueous sample solution at the ideal pH and ionic strength. A 1 mL glass syringe was used to quickly inject a mixture of 500 μL MeCN (disperser solvent) and 80 μL [C_4mim][PF₆] (extraction solvent) into the sample. A fine, stable, hazy emulsion was created by the quick injection. To encourage equilibrium, the tube was vortexed at 2000 rpm for the ideal dispersion period. After that, the emulsion was centrifuged for five minutes at 5000 rpm. Using a 100 μL gas-tight microsyringe, the sedimented IL-rich phase (about 75–80 μL) was collected, diluted with 20 μL MeCN to minimize viscosity, and then 10 μL was injected into the HPLC-PDA system for analysis.

4. Experiments and Results

4.1. Physicochemical Properties of Target Parabens

All four analytes had pK_a values of around 9.0, meaning that they existed mostly in a neutral, undissociated form at pH values much below the pK_a, which greatly favors partition into the hydrophobic IL phase. Due to their shared hydroxyphenyl ester pharmacophore, all four molecules had the same topological polar surface area (tPSA) of 46.53 Å². The steadily rising log P values (1.46, 1.80, 2.29, and 2.71 for MP, EP, PP, and BP respectively) were consistent with the enrichment factors found in the experiments and anticipated growing affinity for [C₄mim][PF₆]. Table 1 compiles pertinent physicochemical characteristics.

Table 1. Physicochemical properties of the four target parabens (data from EPI Suite™/Molinspiration).

Property	MP	EP	PP	BP
Molecular formula	C ₈ H ₈ O ₃	C ₉ H ₁₀ O ₃	C ₁₀ H ₁₂ O ₃	C ₁₁ H ₁₄ O ₃
Mol. weight (g mol ⁻¹)	152.15	166.17	180.20	194.23
Boiling point (K)	548.16	571.04	593.92	616.80
Melting point (K)	359.89	371.16	382.43	393.70
Log P	1.46	1.80	2.29	2.71
pK _a	9.007	8.981	8.970	8.965
tPSA (Å ²)	46.53	46.53	46.53	46.53
CLogP	1.9846	2.5136	3.0426	3.5716
LogS	-1.597	-1.944	-2.218	-2.642
Heat of formation (kJ mol ⁻¹)	-466.51	-487.15	-507.79	-528.43

MP: methylparaben; EP: ethylparaben; PP: propylparaben; BP: butylparaben.

4.2. Optimization of IL-DLLME Conditions

Using a univariate approach, all extraction parameters (pH 7, 80 µL IL, 500 µL MeCN, 10 s dispersion, 0% NaCl, 5000 rpm/5 min centrifugation) were adjusted by changing one factor at a time while keeping the others at their starting values. The optimization response was extraction recovery (ER):

$$ER (\%) = (C_n \times V_n / C_i \times V_i) \times 100 \quad (1)$$

where C_n and C_i are the concentrations in the extract and original sample, respectively, and V_n and V_i are the corresponding volumes. All experiments were carried out in triplicate and mean data were reported.

4.2.1. Effect of Sample pH

The pH range of 2–10 was examined in relation to ER (Figure 1a). The parabens resided mostly as neutral molecules at pH levels substantially below the pK_a values (around 8.97–9.00 for all four analytes; Table 1), which significantly favors their partition into the hydrophobic [C₄mim][PF₆] phase through hydrophobic and π–π interactions. ER values were moderate at pH 2–6 (37–68%) and significantly rose at pH 8, when all analytes showed maximum (MP: 80%; EP: 84 %; PP: 84%; BP: 85%). As the analytes started to ionize toward their phenolate forms above pH 8, ER gradually decreased, decreasing attraction for the IL phase. As a result, pH 8 was determined to be the ideal sample pH.

4.2.2. Effect of Dispersion Time

Vortex periods ranging from 5 to 40 seconds were studied at 2000 rpm (Figure 1b). ER increased from 60–62 % at 5 s to a maximum of 85–89 % at 10 s, with no further substantial improvement noted. The rapid achievement of equilibrium confirmed the excellent mass transfer aided by the fine IL emulsion formed during strong agitation. A dispersion time of 10s was chosen.

4.2.3. Effect of Ionic Liquid Volume

The volume of [C₄mim][PF₆] varied from 5 to 50 µL (Figure 1c). ER reached a plateau at 10 µL for all analytes (MP: 86.2.2 %; EP: 88.4.5 %; PP: 87.2.6 %; BP: 89.1 %). Larger IL volumes did not improve ER but reduced the enrichment factor (EF) by diluting extracted analytes into a larger phase volume. Therefore, 80 µL was selected as the optimal volume to balance recovery and EF. The theoretical EF (10 mL / 0.075 mL $\approx$ 133) was consistent with the experimentally observed range of 120–135.

4.2.4. Effect of Disperser Solvent Type and Volume

MeOH, MeCN, and MeCN:MeOH (1:1, v/v) were the three potential disperser solvents that were assessed (Figure 2a). Due to its better elution strength toward aromatic esters and its capacity to distribute [C₄mim][PF₆] into a homogenous fine emulsion, MeCN provided the highest ER for all analytes (MP: 85%; EP: 88%; PP: 88%; BP: 88%). The ideal disperser solvent volume was determined to be 500 µL of MeCN.

4.2.5. Effect of Ionic Strength and Desorption Time

From 1 to 30 minutes, the impact of desorption time under vortex agitation was assessed (Figure 2b). 10 minutes were chosen since ER reached its peak at the time and then stabilized. Up to 1% NaCl, the addition of NaCl at 0–5.0% (w/v) indicated a modest improvement in salting-out. However, at higher concentrations, there was a slight decline attributed to the increased aqueous viscosity. The ideal ionic strength condition was achieved by adding 1.0% (w/v) NaCl.

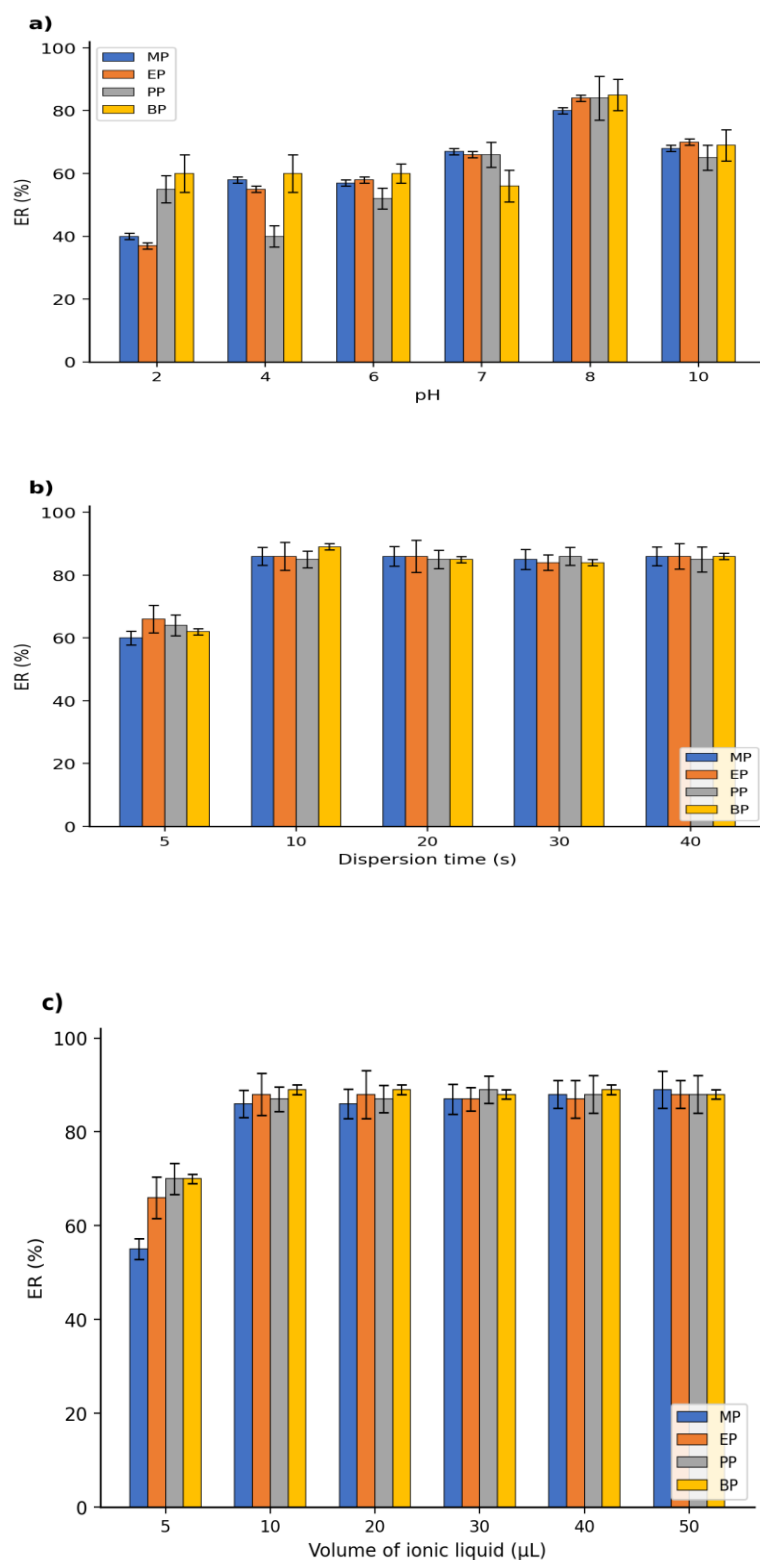


Figure 1. Effect of IL-DLLME extraction parameters on extraction recovery (ER, %) for MP, EP, PP and BP. (a) Effect of sample pH (2–10); (b) effect of dispersion time (5–40 s); (c) effect of [C₄mim][PF₆] volume (5–50 µL). Error bars represent standard deviations (n = 3).

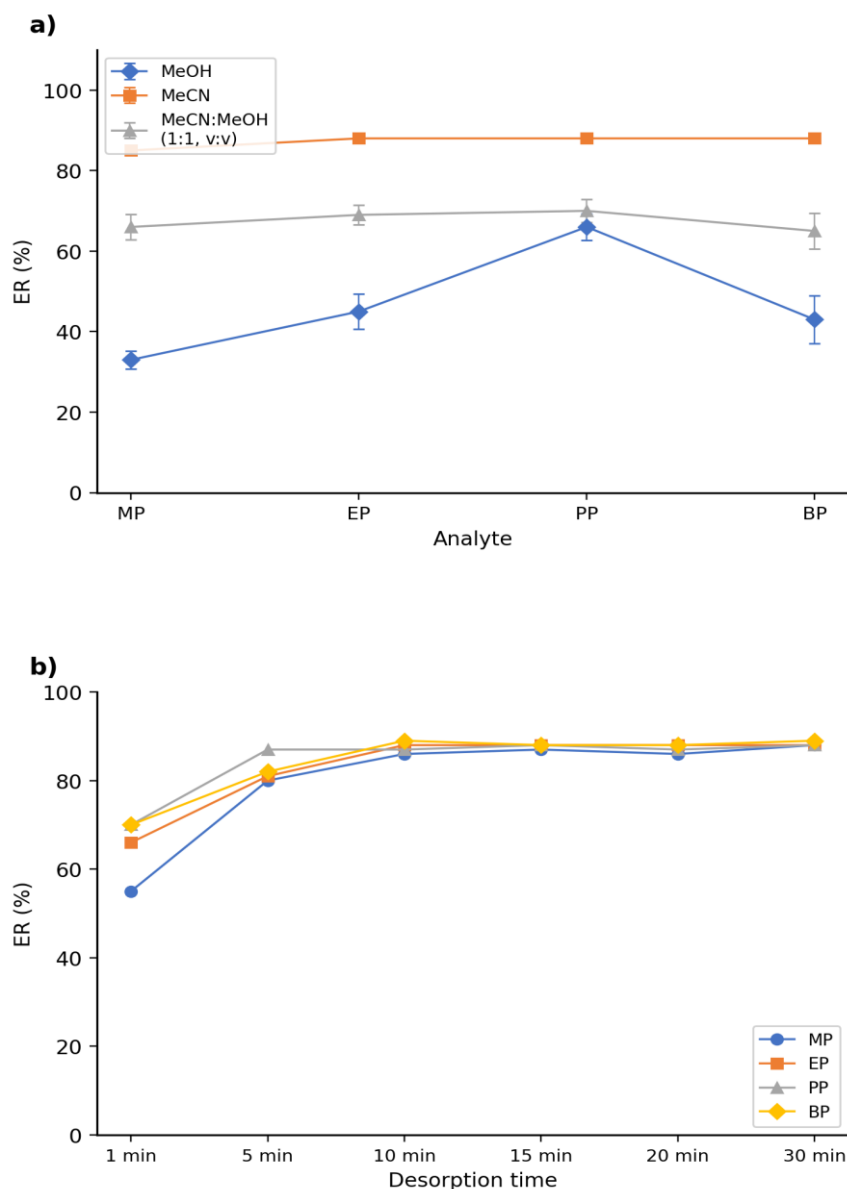


Figure 2. Effect of desorption parameters on ER (%) for MP, EP, PP, and BP. (a) Effect of desorption solvent type (MeOH, MeCN, MeCN:MeOH 1:1 v/v); (b) effect of desorption time (1–30 min). Error bars represent standard deviations (n = 3).

5. Discussion

5.1. Analytical Figures of Merit

Table 2 provides an overview of the optimized IL-DLLME-HPLC-PDA method's analytical performance. The calibration curves had R^2 values of 0.9949–0.9988 and were linear over 100–1000 $\mu\text{g L}^{-1}$. LOD values varied from 0.69 $\mu\text{g L}^{-1}$ (MP) to 1.01 $\mu\text{g L}^{-1}$ (BP), and LOQ values from 1.23 to 1.70 $\mu\text{g L}^{-1}$, derived from S/N ratios of 3 and 10, respectively, on a 50 $\mu\text{g L}^{-1}$ standard exposed to the entire extraction procedure. The repeatability and reproducibility of the intra-day RSD values (2.9–4.8%) and inter-day RSD values (7.6–8.4%; n = 5 over four consecutive days) were satisfactory. Near-quantitative extraction into the IL phase was confirmed by enrichment factors of 120–135.

Table 2. Analytical figures of merit for the IL-DLLME-HPLC-PDA method.

Analyte	LOD ($\mu\text{g L}^{-1}$)	LOQ ($\mu\text{g L}^{-1}$)	Linear range ($\mu\text{g L}^{-1}$)	R ²	Intra-day RSD % ^a	Inter-day RSD % ^a
MP	0.69	1.23	100–1000	0.9949	3.1	7.6
EP	0.85	1.45	100–1000	0.9988	2.9	8.4
PP	0.78	1.35	100–1000	0.9985	4.8	7.6
BP	1.01	1.70	100–1000	0.9955	4.7	7.8

^a Precision at 100 and 500 $\mu\text{g L}^{-1}$ ($n = 5$); values shown are the higher RSD across both concentration levels.

5.2. Reusability Assessment

Over the course of several extraction cycles, the IL extraction phase's reusability was assessed. Before reuse, the collected IL was cleaned twice with 1 mL of pH 7 ultrapure water and dried for 30 minutes at 50°C. The same IL aliquot was used for seven consecutive cycles of analysis of samples spiked at 50 and 100 $\mu\text{g L}^{-1}$. Reliable reuse was confirmed with ER values of 86% through the fifth cycle. Due to the gradual build-up of matrix co-extractants and slight IL breakdown into the aqueous phase, a discernible decrease was seen in the sixth cycle (~75%) and persisted in the seventh. As a result, the IL phase could be consistently reused for up to five cycles without experiencing appreciable performance degradation, significantly lowering solvent usage in comparison to single-use workflows.

5.3. Analysis of Real Toothpaste Samples

Six commercial toothpaste products made in accordance with Section 3.3 were subjected to the optimized procedure. Since every analyte in every unspiked product was below the limit of detection, aliquots spiked at 100 and 500 $\mu\text{g L}^{-1}$ ($n = 3$ each) and put through the entire IL-DLLME process. Table 3 displays the results. Excellent accuracy and consistent precision in the complex toothpaste matrix are demonstrated by ER values of 90.4–101.5% with RSD < 9.7% for all analytes and matrices. Four well-defined paraben peaks at retention periods of 3.62 minutes (MP), 5.16 minutes (EP), 6.54 minutes (PP), and 7.28 minutes (BP) with no conflicting matrix signals can be seen in representative HPLC-PDA chromatograms of an unspiked and a spiked extract (Figure 3).

Table 3. Extraction recovery (ER $\pm$ SD, %; $n = 3$) and enrichment factor (EF) for parabens in six toothpaste products spiked at two concentration levels.

Sample	Analyte	ER (%) at 100 $\mu\text{g L}^{-1}$	RSD (%) at 100 $\mu\text{g L}^{-1}$	ER (%) at 500 $\mu\text{g L}^{-1}$	RSD (%) at 500 $\mu\text{g L}^{-1}$	EF
FT	MP	93.5 $\pm$ 5.8	6.3	97.4 $\pm$ 7.9	8.2	133
	EP	92.1 $\pm$ 5.5	6.0	94.6 $\pm$ 6.8	7.3	130
	PP	99.8 $\pm$ 3.0	3.0	101.5 $\pm$ 4.6	4.6	135
	BP	91.2 $\pm$ 8.6	9.5	96.0 $\pm$ 6.3	6.6	128
WT	MP	90.4 $\pm$ 5.3	6.0	101.5 $\pm$ 8.8	8.7	133
	EP	90.2 $\pm$ 6.5	7.4	96.3 $\pm$ 5.6	5.9	128
	PP	91.0 $\pm$ 5.9	6.5	100.5 $\pm$ 3.5	3.5	133
	BP	90.4 $\pm$ 4.2	4.7	90.8 $\pm$ 8.5	9.6	120
CT	All	90.1–100.2	3.4–9.1	92.0–101.3	3.7–9.5	120–134
ChT	All	90.6–100.5	3.0–8.7	93.0–101.5	3.9–8.9	121–135

HT	All	90.0–99.2	3.7–9.3	91.4–101.0	4.1–9.4	120–133
ST	All	90.3–99.5	3.1–8.8	92.1–101.5	3.6–9.2	122–135

FT: fluoride toothpaste; WT: whitening toothpaste; CT: charcoal toothpaste; ChT: children's toothpaste; HT: herbal toothpaste; ST: sensitive toothpaste. EF: enrichment factor.

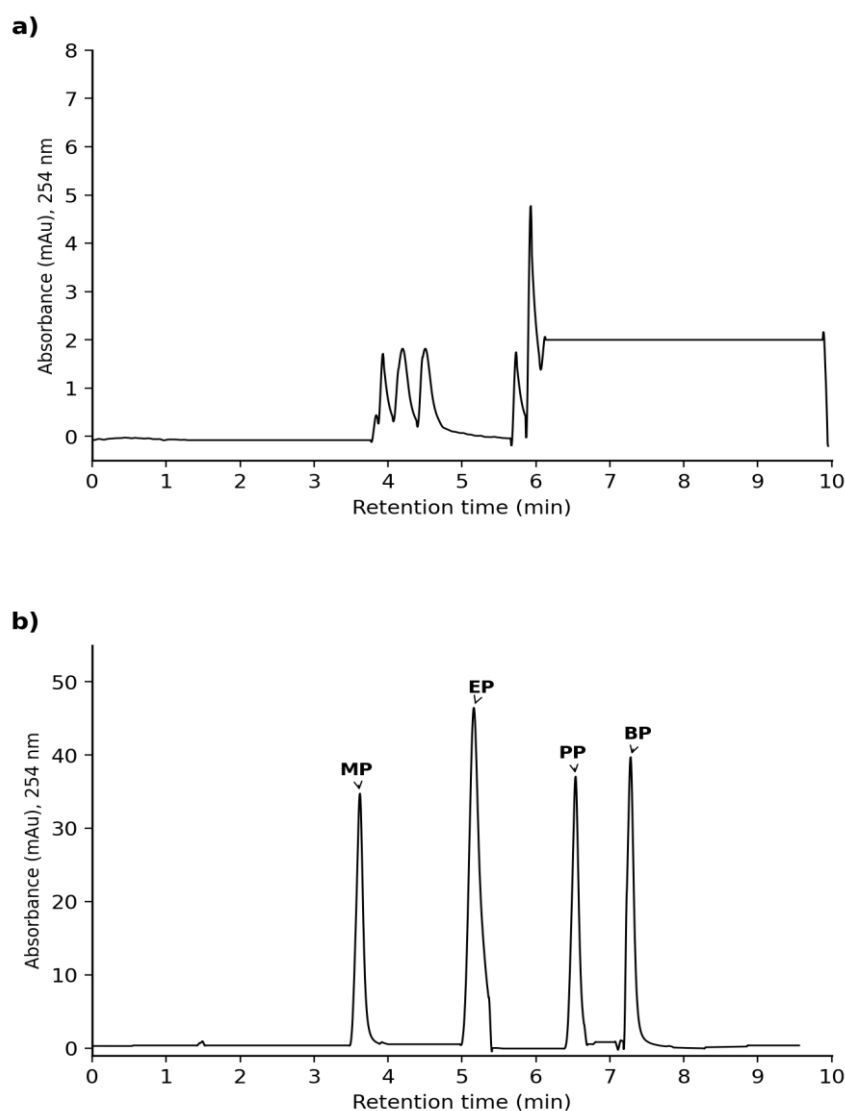


Figure 3. Representative HPLC-PDA chromatograms (254 nm) of toothpaste extracts after IL-DLLME under optimized conditions. (a) Unspiked toothpaste sample; (b) toothpaste sample spiked at $500 \mu\text{g L}^{-1}$ of each paraben. Peak identification: MP = methylparaben ($R_t = 3.62 \text{ min}$); EP = ethylparaben ($R_t = 5.16 \text{ min}$); PP = propylparaben ($R_t = 6.54 \text{ min}$); BP = butylparaben ($R_t = 7.28 \text{ min}$).

5.4. Comparison with Literature

The suggested approach and previously published methods for determining parabens in toothpaste and similar matrices were contrastive in Table 4. That is, the technique yields LODs of $0.69\text{--}1.01 \mu\text{g L}^{-1}$, which were competitive with CPE-HPLC-MS/MS [14] and significantly lower than SPE-LC-UV applied to toothpaste [11]. IL-DLLME offered comparable sensitivity with much simpler preparation—no material synthesis, activation, or magnetic separation—and completed the extraction in around 20 minutes when compared to MSPE processes that required nanomaterial production [16,8]. Minimal reagent usage and less environmental effect were ensured by using only $80 \mu\text{L}$ of ionic liquid and $500 \mu\text{L}$ of MeCN, together with the IL phase's proven reusability.

Table 4. Comparison of the proposed IL-DLLME-HPLC-PDA method with previously reported procedures for paraben determination in toothpaste and related matrices.

Extraction technique	Detection	Matrix	LOD ($\mu\text{g L}^{-1}$)	Recovery (%)	Linear range ($\mu\text{g L}^{-1}$)	RSD (%)	Ref.
SPE	HPLC-MS/MS	Water	12–24	70–115	80–5000	<20	[9]
SPE	LC-UV	Saliva & toothpaste	100–300	86–113	300–5000	<4	[11]
CPE	HPLC-MS/MS	Water	13–38	71–98	9–730	<0.86	[14]
DLLME-CE	CE-UV	Food & milk	100–200	87–103	700–6000	<3.5	[15]
MSPE-MCNPs	HPLC-PDA	Cream products	0.19–0.31	87.9–101.8	20–1000	<8.7	[16]
IL-DLLME	HPLC-PDA	Toothpaste	0.69–1.01	90.4–101.5	100–1000	<8.4	This work

CPE: cloud-point extraction; DLLME-CE: dispersive liquid–liquid microextraction with capillary electrophoresis; MSPE-MCNPs: magnetic solid-phase extraction with magnetic cellulose nanoparticles.

6. Conclusions and Future Work

An IL-DLLME-HPLC-PDA method was developed and validated for the first time for the simultaneous determination of MP, EP, PP, and BP in commercial toothpaste. Systematic optimization of key extraction parameters—sample pH, dispersion time, IL volume, disperser solvent type, ionic strength, and centrifugation conditions—yielded a robust analytical procedure. The method exhibited excellent linearity ($R^2 = 0.9949\text{--}0.9988$), low detection limits ($0.69\text{--}1.01 \mu\text{g L}^{-1}$), satisfactory precision ($\text{RSD} < 8.4\%$), and high enrichment factors (120–135) even in the complex toothpaste matrix. Replacement of halogenated solvents with an ionic liquid extractant, combined with the demonstrated potential for up to five reuse cycles, substantially reduced reagent consumption and environmental impact, making the procedure both analytically powerful and aligned with the principles of green analytical chemistry.

The validated method was successfully applied to six commercial toothpaste products without paraben detection in any unspiked sample, which indicated compliance with current regulatory limits. Future work should explore extending the analyte scope to include additional preservatives (e.g., phenoxyethanol, benzyl alcohol) and investigating alternative bio-based ionic liquids that offer further improvements in environmental profile. Scaling up to a high throughput format and coupling with UHPLC to improve analysis speed are also promising directions.

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8. Declarations

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Conflict of Interest / Competing Interests

The authors declare no conflict of interest.

Ethics Approval and Consent to Participate

Not applicable. This study involved only commercial products and no human participants.

Consent for Publication

All authors have read and approved the final manuscript and consent to its publication.

Data Availability

The datasets generated during the current study are available from the corresponding author upon reasonable request.

Materials Availability

All chemicals and reagents are commercially available as described in Section 3.1.

Code Availability

Not applicable.

Authors' Contributions

F. Siouri: Conceptualization, Methodology, Formal Analysis, Writing–Original Draft. F. Abujaber: Conceptualization, Supervision, Writing–Review and Editing, Funding Acquisition. A. Mubarak: Methodology, Resources, Writing–Review and Editing. All authors have read and agreed to the published version of the manuscript.

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