
Green Sample Preparation: A Critical Review of Techniques Supporting Sustainable Analytical Practices

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Abstract

Green sample preparation is an emerging focus in analytical chemistry, driven by the need to reduce the environmental and health impacts of conventional methods. Traditional sample preparation techniques often require large amounts of toxic organic solvents, high energy input, and extensive handling of samples, resulting in significant waste and safety concerns. In response, sustainable alternatives have been developed that align with the principles of green analytical chemistry, which emphasize efficiency, safety, and environmental responsibility. This review explores recent advancements in green sample preparation techniques that minimize solvent use, reduce energy consumption, and limit hazardous waste generation. Techniques such as solid-phase microextraction, dispersive liquid-liquid microextraction, microwave-assisted extraction, ultrasound-assisted extraction, and supercritical fluid extraction are discussed for their effectiveness, applicability, and environmental benefits. These methods have demonstrated significant improvements in analytical performance while reducing ecological footprints. The scope of this review includes a critical analysis of current methods across various sample types, including environmental, biological, pharmaceutical, and food matrices. Key challenges in method standardization, scalability, and adoption in routine laboratories are also addressed. Overall, this article highlights the transformative potential of green sample preparation as a central element in the shift toward sustainable analytical practices, offering both scientific and environmental advantages.

Keywords: green analytical chemistry, sample preparation techniques, eco-friendly extraction, micro extraction methods, sustainable laboratory practices, green solvents

1. Introduction

1.1 Introduce the problem

Green sample preparation has emerged as a vital component in analytical chemistry, motivated by the urgent need to address the ecological and health impacts of conventional methods. Traditional practices—such as Soxhlet extraction, solid-phase extraction, and liquid-liquid extraction—rely heavily on toxic solvents and energy-intensive procedures, resulting in substantial waste generation and occupational hazards. These inefficiencies contradict global efforts toward sustainability and regulatory compliance. Green Analytical Chemistry (GAC), a discipline that integrates environmental consciousness into analytical processes, underscores the need for low-impact, solvent-minimizing alternatives. Within this context, green sample preparation offers a promising route toward environmentally responsible and efficient analytical workflows.

1.2 Explore importance of the problem

The importance of green sample preparation lies in its potential to mitigate longstanding environmental and health challenges posed by traditional extraction and preparation techniques. The movement is not only about reducing solvent usage or waste but also about fostering sustainable development within scientific practice. Increasing regulatory pressure, growing environmental consciousness, and a shift toward circular economies necessitate analytical methods that are both reliable and eco-friendly. Addressing this problem is crucial, as it enables laboratories and industries to align with green policies, reduce operational costs, and improve workplace safety. This review is thus timely and essential, considering the global shift toward green chemistry applications in pharmaceutical, environmental, biological, and food sciences.

1.3 Describe relevant scholarship

Over the past decade, significant advancements have been made in the development of greener techniques such as supercritical fluid extraction (SFE), microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), and dispersive liquid-liquid microextraction (DLLME). Numerous studies have documented their efficiency and environmental benefits. Płotka-Wasyłka et al. (2016) emphasized the potential of microextraction in reducing hazardous solvent use, while Silva et al. (2023) applied white analytical chemistry principles to validate eco-friendly procedures. Previous reviews have also catalogued innovations in green solvents, miniaturized systems, and biodegradable materials. This paper expands on existing literature by critically examining these methods across diverse sample matrices and highlighting the trajectory of green sample preparation from laboratory experimentation to routine application.

1.4 State hypotheses and their correspondence to research design

This review article does not test a traditional hypothesis but aims to evaluate the hypothesis that green sample preparation techniques offer comparable or superior analytical performance while significantly reducing environmental impact. By synthesizing and comparing experimental

methodologies and outcomes from the literature, the review supports this proposition. The research design involves comparative analysis across key techniques and their practical implementations, with a focus on solvent reduction, energy efficiency, scalability, and method reproducibility. The objective is to identify methods that meet both analytical rigor and sustainability benchmarks.

2. Method

This review synthesizes and critically evaluates various green sample preparation techniques as reported in the scientific literature. No original experimental study was conducted. Instead, this review adopts a qualitative comparative methodology by drawing on peer-reviewed research published between 2015 and 2024.

2.1 Study design

This article follows a structured literature review format. Green sample preparation techniques were categorized into macro and micro extraction methods. The review specifically evaluated solvent reduction, extraction efficiency, reproducibility, energy consumption, and waste minimization. Where applicable, comparative findings were cited from studies that benchmarked green methods against conventional protocols.

2.2 Data sources and selection

Relevant studies were selected using databases such as PubMed, ScienceDirect, and Google Scholar, with keywords including *green sample preparation*, *eco-friendly extraction*, *sustainable solvents*, *SPME*, *DLLME*, and *microwave-assisted extraction*. Only peer-reviewed articles and high-impact reviews were included. Methodologies published in conference abstracts or non-peer-reviewed sources were excluded.

2.3 Operational definitions

Green sample preparation: Any analytical method that reduces or eliminates the use of hazardous solvents and promotes sustainability.

Extraction efficiency: The percentage recovery or yield of the target analyte under optimized conditions.

Green solvents: Solvents classified as environmentally benign due to low toxicity, biodegradability, or minimal environmental impact.

2.4 Summary of methodologies reviewed

The techniques reviewed include:

Supercritical Fluid Extraction (SFE): CO₂-based systems under high pressure and temperature.

Microwave-Assisted Extraction (MAE): Closed-vessel extraction using microwave energy and ethanol/water solvents.

Ultrasound-Assisted Extraction (UAE): Sonication with green solvents for improved mass transfer.

Solid-Phase Microextraction (SPME): Solvent-free fiber adsorption and GC-MS integration.

Dispersive Liquid-Liquid Microextraction (DLLME): Use of minimal volumes of dispersive and extractive solvents.

Pressurized Liquid Extraction (PLE): Accelerated solvent extraction under high pressure and temperature using ethanol or water.

Headspace GC-MS Analysis: For volatile component quantification using minimal sample manipulation.

Each method's procedural steps, instrumentation, and sample matrix were extracted directly from the original study and reported with attention to reproducibility and environmental impact.

2.5 Methodological validity

To ensure reliability, only methods validated by the original authors through recovery studies, reproducibility data (RSD%), or statistical comparison with reference techniques were included in this review.

3. Results

This review synthesized data from peer-reviewed studies published between 2015 and 2024 to evaluate the performance and sustainability of green sample preparation techniques. The following results highlight the environmental benefits, operational efficiencies, and analytical robustness associated with each method.

3.1 Performance and efficiency of green techniques

Across the reviewed studies, all green sample preparation methods—such as SPME, DLLME, MAE, UAE, and SFE—demonstrated considerable improvements in terms of solvent reduction, reduced extraction times, and lower energy consumption compared to traditional techniques. For instance:

Microwave-Assisted Extraction (MAE) reduced extraction time by up to 80% and used up to 60% less solvent compared to Soxhlet extraction (Wang et al., 2025).

Ultrasound-Assisted Extraction (UAE) required minimal thermal input and reduced overall energy consumption while improving analyte recovery from food matrices (Silva et al., 2023).

Supercritical Fluid Extraction (SFE) using CO₂ eliminated organic solvents entirely and achieved comparable yields to conventional liquid-liquid extraction (Herrero et al., 2010).

3.2 Environmental impact and sustainability indicators

Several reviewed studies quantitatively evaluated greenness metrics such as solvent volume, toxicity, waste generation, and biodegradability of reagents. For example:

Ionic liquids and deep eutectic solvents (DES) were shown to reduce toxicity and improve biodegradability compared to chlorinated organic solvents (Prabhune & Dey, 2023).

Green SPE cartridges utilizing biopolymer-based sorbents minimized hazardous waste while maintaining analytical precision.

3.3 Reproducibility and applicability

Techniques such as **Solid-Phase Microextraction (SPME)** and **Dispersive Liquid-Liquid Microextraction (DLLME)** showed reproducible recoveries above 85% across pharmaceutical, environmental, and food sample matrices.

Comparative studies confirmed that miniaturized extraction techniques could be integrated with existing chromatographic systems (e.g., GC-MS, HPLC-DAD) without compromising sensitivity or selectivity.

3.4 Challenges and limitations

Although many green sample preparation techniques showed promising results, challenges persist:

Standardization of method parameters across different laboratories remains a barrier to widespread adoption.

High initial costs for equipment (e.g., microwave or pressurized extraction systems) may limit usage in smaller labs.

Limited solvent polarity range in SFE or ionic liquids can affect extraction efficiency for certain analytes.

4. Discussion

The reviewed studies consistently highlight a shift towards greener methodologies in sample preparation. Wuethrich et al. (2015) demonstrated the potential of solvent-free electric field-driven extraction using hydrogels, setting a precedent for sustainable herbicide analysis in environmental samples. This method aligns with the green chemistry principle of reducing harmful solvent usage and offers significant time savings compared to solid-phase extraction.[26]

Ruiz-de-Cenzano et al. (2015) contributed by comparing digestion methods under green chemistry lenses, identifying microwave-assisted digestion with HNO₃/H₂O₂ as a more sustainable approach than conventional dry ashing techniques.[27]

The review by Psillakis et al. (2017) reaffirmed the role of miniaturized microextraction methods like SPME and DLLME in bioanalysis. These techniques are widely adopted due to their ability to reduce hazardous solvent use, improve extraction efficiency, and integrate seamlessly with analytical instruments like gas chromatography and mass spectrometry.[28]

In 2023, Silva et al. introduced a White Analytical Chemistry (WAC)-based evaluation, using ultrasound-assisted extraction for metal determination in beef. The method's operational simplicity—requiring only 10 minutes, diluted acids, and no external heating—demonstrated

strong environmental and practical benefits, which could be extrapolated to food safety testing.[29]

The comprehensive 2024 review reinforced the increasing adoption of green extraction techniques such as QuEChERS, UAE, MAE, SFE, and SBSE across pharmaceutical and environmental analyses. These methods are not only efficient but also reduce energy consumption and hazardous waste generation.[30]

Plotka-Wasyłka et al. (2016) provided foundational insights into sustainable microextraction practices, emphasizing their role in replacing conventional methods without compromising analytical quality [18].

Table 1 below summarizes notable studies focusing on green sample preparation methods between 2015 and 2024.

Year	Author(s)	Technique	Key Innovation / Focus	Green Chemistry Contribution
2015	Wuethrich et al.	Electric Field-Assisted Extraction	Enrichment/separation of cationic & anionic analytes using conductive hydrogel	Eliminated organic solvents; low-cost and eco-friendly
2015	Ruiz-de-Cenzano et al.	Microwave-Assisted Digestion	Compared MAE with dry ashing for food sample analysis via ICP-OES	Balanced efficiency and environmental impact
2016	Plotka-Wasyłka et al.	Micro Extraction Techniques	Review of sustainable alternatives to conventional preparation methods	Promoted solvent-free or low-solvent techniques
2017	Psillakis et al.	SPME & DLLME	Emphasis on green strategies in pre-chromatographic sample prep	Minimized hazardous solvent use; cost-effective and rapid
2023	Silva et al.	Ultrasound-Assisted Extraction (UAE)	WAC-based evaluation for Fe and Mn detection in beef	Fast (10 min), used diluted acids, no external heating
2024	Future J. of Pharm. Sciences	Multiple (SPE, QuEChERS, etc.)	Review of modern eco-friendly sample prep techniques	Comprehensive overview of sustainable sample preparation tools

Conclusion

The advancement of green sample preparation techniques marks a pivotal step toward sustainable practices in analytical chemistry. The adoption of eco-friendly methods—such as SPME, DLLME, MAE, and SFE—has demonstrated significant benefits by reducing hazardous solvent usage, energy consumption, and waste generation. These methods not only uphold the principles of Green Analytical Chemistry but also enhance laboratory safety and cost-effectiveness. Their growing application in sectors like pharmaceuticals, food analysis, and environmental monitoring underscores their practical relevance and importance. Despite current limitations, including initial costs and method standardization challenges, continued innovation in green solvents, miniaturization, and automation will drive broader implementation. Ultimately, embracing green methodologies ensures that analytical processes remain both scientifically robust and environmentally responsible.

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