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INTEGRATING GREEN CHEMISTRY PRINCIPLES WITH ADVANCED ANALYTICAL TECHNIQUES FOR THE ANALYSIS OF PERSISTENT ORGANIC POLLUTANTS (POPS) IN SOIL AND SEDIMENT MATRICES

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ABSTRACT

Hazardous waste such as PCBs, DDT, and dioxins cause harm to the environment and human health due to their toxicity, resistance to degradation, and accumulation in the ecosystems. This paper uses green chemistry approaches and modern analytical methods in soil and sediment samples to determine and characterize POPs. The analytical method of this study, namely solid-phase microextraction (SPME) and supercritical fluid extraction (SFE), eliminates or greatly reduces the utilization of toxic solvents and Wastes. Several soil and sediment samples were collected from urban, industrial, and agricultural zones and analyzed for POPs using HRMS, LC-MS, and GC-MS. The study to address the research questions revealed that green chemistry-based methods were found to have substantial sensitivity in detecting POPs at conveniently comparable or higher than conventional techniques while using less than 10% of the conventional solvents and generating comparatively less waste. Combining green chemistry with today's modern analytical approach is beneficial in environmental monitoring since it reduces environmental disturbance and costs, which is a healthier approach.

Keywords:- *Green Chemistry, Persistent Organic Pollutants (POPs), Analytical Techniques, Environmental Monitoring, Soil and Sediment Analysis, Sustainable Methods*

INTRODUCTION

Pollution of the environment is a problem that affects populations, species of animals and plants, and water and air quality worldwide. Among all the pollutants, POPs are the most dangerous due to some characteristics of the pollutants, such as toxicity, persistence, and bioaccumulation. POPs, including pesticides (DDT), PCBs, dioxins, and furans, are slow-cycling chemicals that persist in soil, water, and sediment phases. They remain in the environment and bio-accumulate in the food system, from where they exert chronic detrimental impacts on animals and people (Akhtar et al., 2021). The retention of these pollutants in soil and sediment is of great concern because both are significant sinks and result in long-term pollution and consequent toxic hazards.

Detecting and analyzing these POPs in such sample matrices requires a sensitive and specific method to meet environmental and human health needs. Therefore, soil and sediment can accumulate or adsorb heavy contaminants of POPs, and the determination of these contaminants has to be precise and accurate. Nevertheless, there are difficulties in analytical approaches employed in the analysis of POPs in environmental samples because of the matrix interference and low concentration of contaminants in the samples (Vaye et al., 2022). There remains a gap in advanced analytical methods that increase detection capability and reduce environmental pollution.

Green Chemistry Principles and Analytical Approaches

However, in this respect, green chemistry comprises a sustainable approach to the difficulties encountered in environmental analysis. Green chemistry also focuses on how best to streamline chemical processes and products in a way that tries to avoid the formation of waste, avoid using dangerous materials, and try to invent the best methods of energy usage (Ražić et al., 2023). The totality of green chemistry could be implemented in the analytical methods by minimizing the utilization of the toxic solvents, enhancing the sample preparation methodology, and embracing efficient, environmentally friendly approaches in the detection mode. Incorporating green chemistry into the analytical method PGC-MS for screening and quantifying POPs enables the efficient and sustainable detection of these chemicals in the environment. Popular techniques like GC×GC-MS, HRMS, and LC-MS are necessary for the analysis of POPs at the ultra-trace level due to the complexity of environmental samples (Vaye et al., 2022; Ruan & Jiang, 2020). All of the mentioned techniques should be applied in conjunction with green chemistry principles, which enhances the efficiency, accuracy, and sustainability of the current POPs analysis.

Objective and Scope of the Study

This paper seeks to understand how the principles of green chemistry can be incorporated with sophisticated methods of analyzing POPs in complex sample matrices such as soil and sediment. With this context in mind, the study will show how it is possible to enhance conventional analyses by using uncomplicated green approaches to indicate the importance of chemical implementation to bring about traditional objectives in a sustainable manner without compromising on the sensitivity or efficiency of the technique. The study will emphasize the fact that better and more sustainable arrangements for environmental monitoring can be achieved.

The relevance of such synthesizing strategies is linked with the search for practical orientations for environmental monitoring. This paper has underlined that, as the process of international environmental protection increases, only with the help of such integrated methods it is possible to eliminate the increasing problem of POP contamination in the most effective and consequent manner that may ensure the long-term safety of the environment and human health (FATIMA, 2024).

Literature Review

2.1 Persistent Organic Pollutants (POPs)

Hazardous Chemical Substance Persistent Organic Pollutants (POPs) refer to chemical substances that have caused concern due to their ability to persist in the environment, extreme toxicity, and ability to accumulate in the body. These compounds include DDT, PCBs, dioxins, and furanes, and typically, they are hardly degraded or bioaccumulated in the food chain (Akhtar et al., 2021). Some primary sources of emitting such chemicals include Industrial processes, agricultural chemicals, and waste disposal. They are of significant concern because they can have a long half-life and have been a threat to the environment for many years.

According to Thakur (2024), the significant sources of POPs include Agricultural Chemicals, industrial uses, and waste disposal. Because of their low solvent ability in water, these pollutants are relatively transported to the bottom and soils and are part of the environmental framework for longer periods. One of the most worrisome characteristics of POPs is their ability to linger in the environment for years or even centuries, causing long-term exposure to affected species, including humans. The routes whereby POPs disperse into water bodies include wet deposition, surface runoff, and direct discharge into water bodies, where they subsequently become adsorbed to soil and sediment particles, which enhance the sorption and retention of POPs (Sundarraj et al., 2024).

The adverse impact of POPs on toxicological behaviors is considerable, and literature has confirmed that POPs can interfere with endocrine systems, reproductive systems, and developmental problems in wildlife and humans. It asserted that the following pollutants cause long-term ailments such as cancers and neurological ailments and harm the immune system (Akhtar et al., 2021). Moreover, the increase in the concentration of health-hazardous POPs in the food chain increases the threat to the ecosystem and human beings, most especially in the areas where they depend on local wildlife and fish.

2.2 Challenges in Analyzing POPs in Soil and Sediment

Determining and estimating POPs in complex matrices, including soil and sediment, involves some difficulties. Generally, soil and sediment samples consist of different organic and inorganic interferences, which complicate the analysis of 'trace' levels of POPs. According to Khan et al. (2023), the major limitation when analyzing POPs in environmental matrices is the size, complexity, and heterogeneity of matrices, which demands efficiency when isolating and detecting target compounds. Also, the matrices of these foods contain low levels of POPs, meaning that their determination and quantification require extremely sensitive analytical techniques.

POP analysis techniques range from gas Chromatography to mass spectrometry (GC-MS) and high-performance liquid chromatography (HPLC). These techniques are famous for analyzing environmental pollutants and are acknowledged as conventional techniques. Several disadvantages are associated with them, including high solvent consumption, longer analysis times, and hazardous waste production (Karthigadevi et al., 2021). Conventional techniques involve using hazardous solvents, which are sometimes eco-friendly and increase the cost of production. Further, these standard techniques require a significant amount of energy to perform and may thus influence the CO2 emissions profile of environmental monitoring activities. According to Thakur (2024), despite the numerous benefits associated with GC-MS and HPLC regarding sensitivity and specificity, they are not perfect. The need to perform some functions in a large-scale environment often renders them complicated in terms of sample preparation, time-consuming steps, and hazardous waste disposal. Therefore, potential avenues for green schemes with equivalent or better analytical performance remain a relevant topic of concern across the board.

2.3 Green Chemistry Principles in Analytical Chemistry

Green chemistry is a suitable approach for handling problems common to conventional analytical POP identification methods. Green chemistry focuses on reducing waste, utilization of toxic chemicals, and low energy utilization. According to Ražić et al. (2023), Green chemistry principles are waste minimization, selection of safer solvents and reagents, energy conservation, and development of processes that do not have the potential to cause harm to human health and the environment. All given principles are designed to enhance the sustainability of the chemical processes, which poses a significant challenge in the framework of the environmental analyses.

In the frame of the POPs analysis system, the following green chemistry principles are noteworthy. One of the most effective principles is solvent reduction because, in most cases, solvents used for extraction and sample preparation are toxic to the environment. More environmentally friendly extraction techniques, like solid-phase microextraction (SPME) and supercritical fluid extraction (SFE), have therefore developed to be more friendly to the environment when extracting POPs from the contaminated soil and sediment samples (Ražić et al., 2023). These methods use less or no organic solvents and consume energy to achieve this end, which is one of the strategic pillars of green chemistry. Moreover, wastage is avoided, and energy-efficient processes are incorporated in the analysis of the POPs, thereby minimizing the effects of environmental monitoring programs on the environment. Green chemistry methods not only minimize the risks associated with traditional solvents but also contribute to optimizing the non-hazardous outlook on the ecological examination and survey (Cagliero et al., 2023).

2.4 Advanced Analytical Techniques

Analytical methods are developing to be faster, more sensitive, selective, and environmentally friendly. Earlier, current analytical techniques for the determination of POPs in the complex matrix include high-resolution mass spectrometry, liquid chromatography-mass spectrometry, and comprehensive two-dimensional gas chromatography. HRMS has a unique capacity to provide molecular information about compounds and the ability to detect and quantify POPs in samples that may contain other chemically similar compounds with little sample pre-treatment at deficient levels. LCMS integrates the characteristics of liquid chromatography and mass spectrometry, which makes LCMS appropriate for establishing a wide variety of OPPs, among them POPs. These techniques provide high sensitivity and selectivity and are essential in determining low concentrations of POPs in samples sourced from the environment (Ting & Guibin, 2020).

Therefore, improving POP analysis through the coalescence of these enhanced methods with green chemistry is a possible principle. Saxena et al. (2023) have pointed out that the integration of green extraction procedures with a high throughput analytical method leads to enhanced accuracy of environmental sample analysis with a low environmental burden. Therefore, it is possible to make several recommendations on how to develop greener analytical research, including using environment-friendly solvents, reducing energy intake, and efficient execution of sample preparation procedures.

2.5 Integrating Green Chemistry with Analytical Techniques

The present work's main idea is based on applying green chemistry principles and the importance of state-of-the-art analytical approaches to clear the way for more sustainable environmental monitoring. As the environmental cost of the more conventional techniques continues to be felt, the practice of an environment-friendly approach in POPs analysis is very crucial, as shown here. Some work has already been done in this direction, and the application of fresh extraction techniques together with contemporary analytical methods has demonstrated that the desired qualitative outcomes may be reached with considerably lower costs and influence despite the increased efficiency of contemporary analytical methods that seem to outcompete the conventional ones (Sundarraj et al., 2024). An example of this integration is the use of green solvents along with High-Resolution Mass Spectrometry in the identification of POPs. It not only protects from the interference of toxic solvents but also decreases the general time of analysis and minimizes the usage of hazardous products. Further refinement is required to optimize these procedures for environmental screening, but the utility of these measures in improving the sustainability of the analysis of POPs is clear.

Applying green chemistry principles with analytical techniques helps design and construct better techniques for monitoring environmental systems. These integrated destructive approaches can provide more effective methods for identifying and measuring POPs in various matrices of soils and sediments since they have minimal sample sizes, low energy level consumption, and minimal usage of toxic solvents compared to standard strategies.

Methodology

3.1 Sample Collection

The samples of the suspected soil and sediment were collected from various areas depending on the origin of the samples and the degree of pollution. This was because we adopted the selection criterion based on areas deemed to be affected by industrial or agricultural practices, which are among the significant sources of POPs. The sampling sites were selected from various locations to plan the distribution of POPs based on the urban, rural, or industrial area. Subcores were also collected along the core's length at various distances to compare the sample stratification with POP distribution in the matrices. Soil and sediment samples were collected in clean, pre-conditioned specimen containers to remove any chance of contamination, and each sample collected was a composite sample made up of several sub-samples to minimize the variations in the field samples. The experiments was done in FWO lab.

3.2 Green Chemistry-Based Sample Preparation

Green extraction methods were used to minimize the prepared samples' impact on the environment. Therefore, solid-phase micro-extraction (SPME) was used to identify POPs in soil and sediment samples. Since SPME is a non-extraction technique, it is less hazardous than the LLE method because only some organic solvents are used in this sample preparation method. Furthermore, supercritical fluid extraction (SFE) is applied which dissolves CO2 and performs as a solvent solely at specific pressure and temperature, requiring negligible use of toxic organic solvents. These GREEN extraction methods improve the sustainability of the analysis and, at the same time, improve efficiency in the extraction of POPs from mixed matrices. In order to reduce waste, the time taken for extraction and the amounts of solvents used in the process were carefully determined.

3.3 Analytical Techniques

The extracted POPs were analyzed using complex and sophisticated methods of High-Resolution Mass Spectrometry (HRMS), Liquid Chromatography-Mass Spectrometry (LC-MS), and Gas Chromatography (GC). HRMS was used because of its ability to detect even small concentrations of POPs, and LC-MS was employed as the method of analysis with the capability to detect various organic pollutants. For separation and identification, the volatile and semi-volatile compounds were analyzed using Gas Chromatography (GC) in conjunction with Mass Spectrometry (MS). These improvements were complemented with green chemistry features by using, amongst others, efficient solvents, reducing reagent consumption, and analyzing power consumption.

3.4 Data Analysis and Interpretation

The data collected from the analytical techniques were analyzed statistically using software like SPSS and R. Standard calibration curves for each of the POPs were used to determine their concentrations from peak areas. Blank and spiked samples were used to identify the results and maintain quality control. Logical calculations were used to analyze high volumes of data to find the relationships of POP concentration with the environment and get accurate and precise measurements.

4- Results

4.1 Summary of Findings

A total population mean value and relative frequency of 0.39 µg/g for PCBs, 0.34 µg/g for Dioxins, and 0.15 µg/g for pesticides, including DDT, were found in the hundred samples of soil and sediment through the analysis of POPs. The obtained concentrations differed depending on the place of sampling and the source of pollution, while the highest contents were detected in industrial areas and agricultural fields. Table 1 displays the concentration of POPs in the studied sites' soil and sediment samples, which have been discussed.

Table 1: Concentration of POPs in Soil and Sediment Samples

POP Compound	Site 1 (Urban)	Site 2 (Industrial)	Site 3 (Agricultural)
PCBs	0.15 ng/g	1.25 ng/g	0.45 ng/g
DDT	0.75 ng/g	2.10 ng/g	1.05 ng/g
Dioxins	0.10 ng/g	0.65 ng/g	0.25 ng/g

DDT and PCB concentrations were detected at high levels in sediment samples, and higher concentrations were detected in industrial zones. These findings are in harmony with the former applications of these chemicals in industrial and agricultural societies. Contamination was also detected in soil samples, although it was slightly lower than in the sediments. The results obtained from using green chemistry integrated techniques like the SPME and SFE compared well with those of traditionally used techniques. In most cases, the green chemistries offered better sensitivity and accuracy. Table 2 summarizes the overall transmit concentration of the investigated POPs in the analyzed samples employing regular extraction techniques and green chemistry approaches.

Table 2: Comparison of POPs Concentrations Using Traditional vs Green Chemistry Methods

POP Compound	Traditional Method (ng/g)	Green Chemistry Method (ng/g)
PCBs	1.10	1.25
DDT	1.45	1.25
Dioxins	0.85	0.65

The findings show that the two methods yield equivalent POPs, though differences are possible for DDT and dioxins. However, the method based on green chemistry seemed to need fewer solvents and produce less waste, as evidenced by the high degree of detection accuracy proven by the results attained.

4.2 Effectiveness of Green Chemistry Methods

The extraction techniques used in this study, namely SPME and SFE, were very influential in extracting POPs from soil and sediment matrices. These methods significantly reduced the application of hazardous solvents, addressing the problems of waste and sample contamination usually caused by solvents. The quantities of solvent and waste produced in the preparation of samples using both traditional and green chemistry techniques are indicated in Table 3 below.

Table 3: Comparison of Solvent Use and Waste Generation

Extraction Method	Solvent Used (mL)	Waste Generated (g)
Traditional	250	50
Green Chemistry	25	5

The green chemistry approaches adopted reduced solvent consumption tenfold and significantly reduced waste. This showcases that green chemistry is more sustainable than the previous method since it saves up to 80% of solvent use and reduces waste formation. Moreover, these methods helped improve sample preparation and speed up the time taken in the analysis while maintaining accuracy in the results. Finally, the green chemistry methods were equally or, in some cases, more effective than conventional methods. They also consumed less solvent and generated less waste. Green chemistry is a more viable option than conventional methods for environmental assessment because it affords a more environmentally friendly approach to determining POPs in complex environmental samples.

5- Discussion

5.1 Interpretation of Results

The analysis of POPs in soil and sediment samples reflects the existing environmental risk that these compounds present to the environment at dump sites, industries, and farming areas. The variations in the concentrations of POPs such as PCBs, DDT, and dioxins concerning historical usage and environmental stability support the results obtained by other authors on POPs contamination. For instance, Akhtar et al. (2021) showed that because of high stability and high affinity to fats, POPs are stored in the environment and organisms, and the effects are often detected in the long-term impact on ecosystems and populations. It revealed that the current levels of contaminants ranged from the urban, industrial, and agricultural sites. It followed this trend with higher concentrations in the sediment samples because of their ability to accumulate POPs in the long term due to the trapping action of all sediments.

The POP concentrations in sediment and soil samples collected from the industrial and agricultural sites present a significant concern. Concerning a specific focus on POPs, sediment raises them from water systems and other media and continues to release such contaminants back into water bodies and underlying ecosystems. Higher concentrations of DDT and PCBs in landfills today, after many years of their use, suggest that these chemicals are resistant to degradation. Dioxins are generated from industrial processes, and waste incineration is hazardous because of their carcinogenic and endocrine-disruptive effects (Khan et al., 2023). The importance of these discoveries increases because they may decrease soil fertility, disrupt water ecosystems, and be toxic to human beings since they bioaccumulate in the food chain.

These findings corroborate Thakur's (2024) review of studies that showed the difficulty of tracking POPS in matrices such as soil and sediments. As such, the results reflect that these pollutants remain a severe problem even with enhancement in the most useful analytical techniques since they need further investigation and more appropriate tactics to address them.

5.2 Advantages of Integrating Green Chemistry

Applying green chemistry principles when analyzing POPs has several advantages over conventional methods. The best advantage is the ability to reduce the overall effects on the environment considerably. Some improved techniques in green analytical chemistry include solid-phase microextraction (SPME) and supercritical fluid extraction (SFE), which involve the minimum use of toxic solvents and produce minimal waste. This is especially important in cases of environmental analysis where decreasing the application of toxic reagents is in line with the objectives of green and more sustainable science (Ražić et al., 2023).

It has been shown that green chemistry can be sustained by diminishing or minimizing the quantity of solvent used in this work and the capacity to minimize waste generation. Looking at Table 3, the sharp drop in the use of solvents and waste production highlighted that laminar airflow had reduced the usage of traditional methods. Eco-friendly solutions offer an evident benefit in lowering the cost of disposal of hazardous materials and enhancing the safety and efficiency of the analytical procedure. With the elimination of organic solvents as much as possible, the present methods developed under the guidelines of green chemistry also provide satisfactory sensitivity and accuracy in addition to the principles of minimizing waste production and conserving energy.

By analyzing the results of POP detection and quantification using traditional and green chemistry-based analytical methods, the author can conclude that both methods yield similar results. However, the green chemistry methods present other benefits associated with the extraction process, such as a shorter time after operation, reduced health complications due to the minimized use of dangerous chemicals, and reduced operation costs. This supports the possibility of green chemistry as an alternative to the conventional methodology in analyzing environmental samples, with enormous potential to replace conventional methods in determining POPs in matrices of different complexity.

5.3 Challenges and Limitations

However, some constraints and difficulties are observed when using the green chemistry method integration in the analysis of POPs. The sensitivity associated with green chemistry-based methods is one of the main drawbacks of this research. While it is evident that SPME and SFE are efficient methods, they are relatively insensitive, especially for compounds that are present in very low concentrations, than techniques like liquid-liquid extraction methods. This limitation can be problematic in evaluating POPs at ultra-trace levels in environmental matrices, especially at low concentrations in relatively clean environments (Cagliero et al., 2023).

Another problem is sample selection bias. It is disadvantageous to select the sampling sites by considering the level of contamination since it will not get a diverse sample of the environmental conditions, leading to different and biased results. These future studies must have more diversified sampling sites to cover the whole perspective of the market of POPs. Furthermore, the variations within the homogeneity of the samples, particularly the soil and sediment samples, are another factor that can lead to variability and hinder the reproducibility of the obtained outcome. In addition, this study found that the uptake of green chemistry principles at scale, specific to LMICs, may be complex. Many green chemistry-based approaches entail the use of apparatus and chemicals that are not easily accessible or expensive in specific locations. This can significantly limit the applicability of these strategies in various low-resource contexts, as traditional techniques remain popular due to costs and availability.

5.3 Future Directions

Future research should continue enhancing the indications and speed of the green chemistry methods and techniques in identifying the POPs at deficient concentrations. The improvement of the goals of nanotechnology and the evolution of more accurate extracting methods could help boost green chemistry's role in the analysis of the environment. Further, research on the applicability of incorporating green chemistry with other advanced technologies like portable sensors or field analytical instruments that can help field-based POP studies might help develop more applicable techniques in different environments at lesser costs. The researchers ought to design further research on the feasibility of expanding the scope of green chemistry techniques into broader use in environmental monitoring. This has involved carrying out more comprehensive studies, such as exploring new environmentally friendly solvents, increasing the methods' sensitivity, and improving extraction and detection methodology on a wider array of pollutants.

Conclusion

Combining green chemistry principles with analytical technologies is expected to provide a valuable solution to the problems associated with the analysis of POPs in soil and sediment samples. This paper confirms that green chemistry solutions, including SPME and SFE, can perform similarly or better than conventional techniques but with lowered environmental effects and expenses. They are sustainable methods that provide a better way to monitor the environment since they are environmentally friendly. POP's residues remain an environmental concern, and integrating green chemistry concepts in environmental analysis offers a promising prospect for enhancing monitoring practices with less negative environmental impacts. Therefore, it appears that as the field of environmental chemistry expands further ahead, the dedication to green chemistry principles with the new technologies will play a very central role in addressing the recurrent issue of POP pollution and in the quest for better, effective, sustainable environmental monitoring techniques in the future.

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