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A Study on the Current Status of Microplastic Analysis in the Air Using FT-IR

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Abstract

Purpose: To collect and analyze microplastics in the atmosphere, a preprocessing method is necessary. Currently, reliable preprocessing methods recognized in academia include oxidation treatment and density separation. **Research design, data and methodology:** Oxidation treatment is a process for removing organic matter in the sample. By adding 20 mL of H_2O_2 and 20 mL of Fenton oxidation solution to the sample and heating it at 60°C for 2 hours, the organic matter in the sample is oxidized and removed. Density separation is a process for removing inorganic matter in the sample. By adding 40 mL of $ZnBr_2$ density separation solution to the sample and allowing it to settle in a separatory funnel for 24 hours, the supernatant is collected for filtration. At this stage, the stopcock at the bottom of the separatory funnel is opened to remove the precipitated inorganic matter based on density differences. **Results:** Additionally, before applying these preprocessing methods, a resuspension process is required to detach microplastics from the actual sample. This is achieved through sonication for 10 minutes. **Conclusions:** The objective of this study is to investigate the current status of FT-IR analysis in Korea and to determine the optimal analytical method.

Keywords : Microplastics, Spectroscopic Analysis, FT-IR

JEL Classification Code : Q38, Q48, Q51

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1. Introduction

Atmospheric microplastics refer to plastic fragments smaller than 5 mm, which are generated from plastic waste through erosion, friction, and other mechanical processes.

Common components of microplastics include high-density polyethylene (HDPE), low-density polyethylene (LDPE), polypropylene (PP), polystyrene (PS), polyethylene terephthalate (PET), and polyvinyl chloride (PVC).

Larger microplastics tend to settle in the ocean, where they circulate within the marine ecosystem and eventually enter the human body through the food chain.

On the other hand, atmospheric microplastics exist as airborne particles smaller than 10 micrometers, comparable in size to fine dust (PM). These particles are carried by the wind and transported across various regions.

Microplastics of PM10 and PM2.5 sizes can be inhaled into the human respiratory system, penetrating deep into the lungs and potentially causing various respiratory diseases. Therefore, raising awareness about their risks is crucial.

Thus, to enhance awareness of the hazards of atmospheric microplastics and establish standards for assessing their impact on human health, this study investigates the current status of atmospheric microplastic analysis using FT-IR (Fourier Transform Infrared Spectroscopy) and highlights the importance of preprocessing methods in this analytical process.

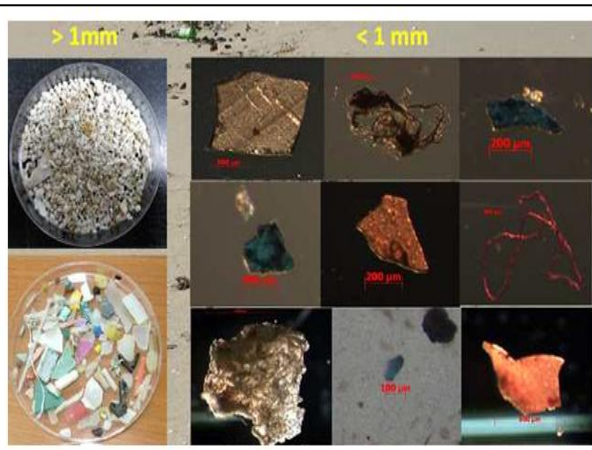

Figure 1: Microplastic Characteristics by Size

2. Literature Review

Currently, analytical methods for microplastics being studied in academia include FT-IR, Raman spectroscopy, and TOC analysis.

These methods can be categorized into spectroscopic analysis and thermal analysis. Additionally, based on a sample size threshold of 45 micrometers, FT-IR and Raman spectroscopy are used for different size ranges.

In this study, we focus on FT-IR analysis, which is primarily used for relatively larger microplastics.

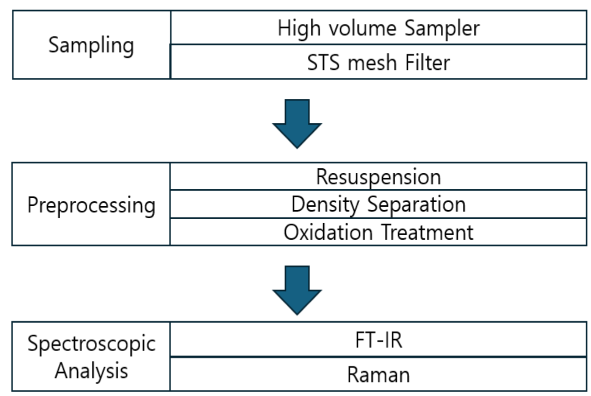

Figure 2: the procedure for spectroscopic analysis of airborne microplastics

2.1. FT-IR

The Fourier transform-infrared spectrophotometer is one of the representative instruments used for analyzing microplastics. It measures the absorption or reflection of infrared radiation in the infrared range of the sample, which varies depending on the molecular structure of the material. The absorption or reflection characteristics of infrared radiation at specific frequencies differ according to the internal molecular structure of the material.

Fourier transform (FT) is a mathematical technique used to decompose a signal into its frequency components. When a time-domain signal is subjected to a Fourier transform, it becomes a frequency-domain function.

For microplastic analysis, a Micro Fourier Transform Infrared Spectrometer (Micro FTIR) equipped with an optical microscope is used. This allows for the detailed analysis of small particles, enabling the identification of specific polymers that make up the microplastics by measuring the infrared absorption spectra.

Figure 3: Fourier transform-infrared spectrophotometer

2.2. Preprocessing Methods

To analyze airborne microplastics using spectroscopic instruments, pre-treatment is essential to remove interfering substances.

Currently, well-known pre-treatment methods in academia include oxidation treatment and density separation.

- Oxidation Treatment:
This process removes organic materials from the sample. A mixture of 20 mL of hydrogen peroxide (H_2O_2) and 20 mL of Fenton's reagent is added to the sample and heated at 60°C for 2 hours. This treatment oxidizes and removes the organic matter present in the sample.

- Density Separation:
This method removes inorganic materials from the sample. A 40 mL solution of zinc bromide ($ZnBr_2$) is added to the sample. After 24 hours, the sample is allowed to settle in a separating funnel. The supernatant is collected for filtration. During this process, the stopcock at the bottom of the separating funnel is opened to remove the inorganic materials that have settled at the bottom due to the density difference.

Additionally, prior to these pre-treatment methods, it is necessary to re-suspend microplastics from the sample for efficient separation. This is achieved by sonication, which involves using ultrasonic waves for 10 minutes to agitate and detach the microplastics from the sample.

These pre-treatment steps ensure the removal of organic and inorganic interference, making the spectroscopic analysis of microplastics more accurate and reliable.

3. Research Methods

The pre-treatment process for microplastic analysis using FT-IR is carried out in the following steps:

The pre-treatment process consists of re-suspension, density separation, and oxidation treatment. During the pre-treatment stages, certain steps may be omitted based on the experimenter's judgment, depending on the requirements of the analysis.

Since microplastics are susceptible to contamination, all pre-treatment steps are conducted while sealing the sample with aluminum foil or similar materials, except when reagents are added. This ensures that contamination is minimized throughout the process.

3.1. Re-suspension

The sample is treated with sonication for 10 minutes using ultrasound. This re-suspension step helps detach the actual microplastics from the filter.

3.2. Density Separation

A 20 mL solution of density separation ($ZnBr_2$) is added, and the filter is treated with ultrasound for 10 minutes to detach the microplastics adhering to the filter surface. After the sonication, the sample is left to settle in a separating funnel for 24 hours.

The density separation solution is carefully poured into the funnel to avoid disturbing the settled precipitates. The supernatant is then separated. The collected supernatant is filtered through a 5 µm SUS filter and stored in a Petri dish.

Lastly, if salt is formed when the density separation solution comes into contact with hydrogen peroxide or distilled water, sulfuric acid is diluted to create a mild acid, which is then used for filtration.

3.3. Organic Matter Oxidation

Since salts may form when oxidizing agents are mixed with distilled water, oxidation treatment is carried out after density separation. The filter, after density separation, is transferred to a cleaning container. Then, 20 mL each of hydrogen peroxide and Fenton's solution are added, and the mixture is heated at 70°C for 2 hours. After the heating process begins, additional 20 mL of hydrogen peroxide and Fenton's solution are added after 1 hour.

Once the 2-hour heating is complete, the sample is subjected to multi-stage filtration using a 20 µm - 5 µm

SUS filter to classify out larger interfering particles that could hinder microplastic analysis.

4. Results and Discussion

Based on the experimental results and comparison with various studies on airborne microplastic analysis, the following conclusions were drawn:

Without Pre-treatment: The presence of insect remains, seeds, and inconsistent interfering substances made FT-IR analysis impossible.

Re-suspension During Pre-treatment: FT-IR analysis was possible after re-suspension, but when attempting to analyze smaller microplastic fragments, interference from other substances prevented successful analysis.

Density Separation During Pre-treatment: Even though only inorganic materials were removed, FT-IR analysis was still possible, suggesting that this step helped reduce interference.

Oxidation Treatment During Pre-treatment: This process effectively removed interfering substances, particularly organic materials, which significantly impacted the analysis. As a result, FT-IR analysis became more effective and reliable.

Therefore, When only oxidation treatment was applied to the sample, sufficient removal of interfering substances was achieved, allowing FT-IR analysis without any issues. However, for effective detachment of microplastics from real-world airborne samples, a re-suspension step during pre-treatment is essential.

5. Conclusions

Based on the comparison between the airborne microplastic experiments conducted in this study and the current literature being researched in academia, the following conclusions were drawn:

Interfering Substances in Samples: Due to the high presence of organic and inorganic materials in the samples, without physical treatment (density separation) or chemical treatment (oxidation), the analysis is not feasible because of the interference caused by these substances.

Importance of Oxidation Treatment: The removal of organic matter through oxidation treatment is essential as it significantly reduces interference. Inorganic materials, on the other hand, can be somewhat removed through multi-stage filtration, although some loss may occur during density separation.

Sample Preservation During Pre-treatment: The higher the preservation rate of the sample after pre-treatment, the more samples can be analyzed. This highlights the importance of controlling and improving the process during pre-treatment to ensure high recovery rates of the target microplastics.

Material Compatibility for Sample Handling: It is crucial to avoid using plastic or similar materials that could interfere with the microplastic measurements. Equipment in direct contact with the samples or reagents should be made from materials such as stainless steel, aluminum, or glass to avoid contamination.

Precautionary Measures During Experimentation: Factors such as contamination during cleaning procedures (e.g., cleaning glassware and funnels) can introduce errors into the results. Therefore, guidelines are needed to ensure not only appropriate pre-treatment methods but also thorough cleaning of experimental tools and preventing contamination due to environmental factors.

Plastic Contamination in Laboratory Equipment: Since microplastics can adhere to the surfaces of glassware used in pre-treatment processes, a cleaning procedure to detach these particles is necessary. It's important to conduct cleaning using distilled water in a glass beaker to avoid contamination from plastic cleaning instruments.

Future Research: By incorporating the improvements identified in this study, future research on pre-treatment processes for airborne microplastics will lead to more accurate analysis with higher recovery rates. This will provide a more effective FT-IR analysis method for microplastic detection in the air.

By refining the pre-treatment methods and ensuring strict procedural controls, this study provides a foundation for improving FT-IR analysis techniques for airborne microplastics in future research.

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