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A Study on the Current Status of Microplastic Analysis in the Air Using Raman

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Abstract

Purpose: To collect and analyze microplastics in the atmosphere, a pretreatment method is necessary to remove interfering substances. **Research design, data and methodology:** Currently, the most reliable pretreatment methods widely used in academia are oxidative treatment and density separation. These methods vary depending on the sample size and the analytical instruments used, and each requires different considerations and processes. **Results:** Oxidative treatment, for instance, is used to remove organic matter from samples. This typically involves adding 20 mL of hydrogen peroxide (H_2O_2) and 20 mL of Fenton's reagent to the sample, then heating it at 60°C for two hours. During this process, the organic matter in the sample is oxidized and removed. Density separation, on the other hand, is used to remove inorganic substances from the sample. In this method, 40 mL of a $ZnBr_2$ (zinc bromide) density separation solution is added to the sample. The mixture is then left to settle in a separatory funnel for 24 hours. After settling, the supernatant is collected for filtration, and the bottom of the separatory funnel is opened to discharge the inorganic matter that has precipitated due to the difference in density. **Conclusions:** Thus, the aim of this study is to investigate the current status of analytical methods using Raman spectroscopy in Korea and to identify the optimal pretreatment method suitable for analyzing substances as small as microplastics.

Keywords : Microplastics, Raman Spectrometer, Analysis

JEL Classification Code : R20, Q20, Q50

1. Introduction

The range of fine dust commonly known to the public includes PM10 and PM2.5, with particle sizes of 10 µm and 2.5 µm, respectively, referred to as fine dust and ultrafine dust.

Microplastics also come in various types, with representative examples including polypropylene (PP),

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polystyrene (PS), polyethylene terephthalate (PET), high-density polyethylene (HDPE), low-density polyethylene (LDPE), and polyvinyl chloride (PVC). Among microplastic particles, some also fall within the particle size range of fine dust.

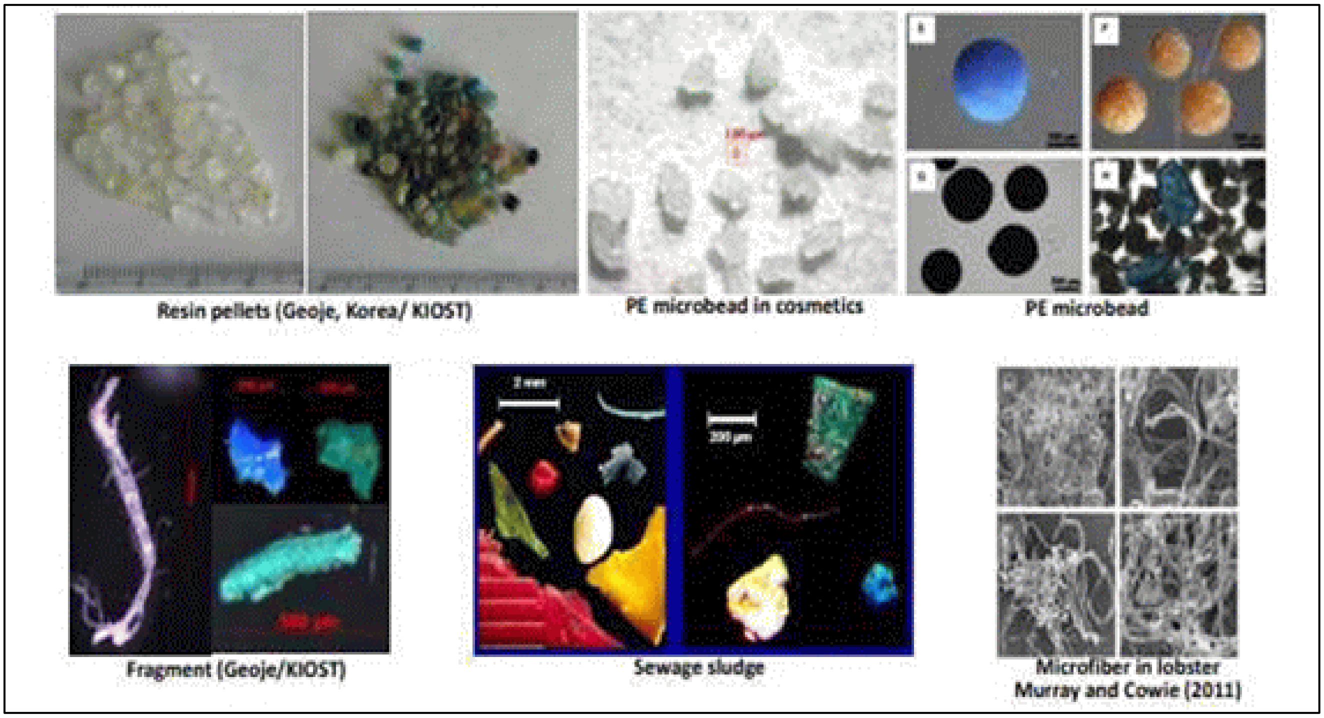

Figure 1: Various appearances of microplastics

While microplastics in the atmosphere are generally classified as particles smaller than 5 mm, the actual airborne microplastics are typically found in much smaller size ranges.

However, this categorization is not clearly defined, and while the public is relatively well aware of fine dust, awareness of the risks associated with microplastics remains low.

In general, microplastics tend to remain airborne and travel long distances. Those capable of affecting the human respiratory system are mostly found in size ranges below 1 μm.

Therefore, to analyze smaller microplastic particles, it is necessary to establish Raman spectroscopy as an analytical method and to standardize the pretreatment processes involved in the analysis.

2. Literature Review

Currently, the main spectroscopic methods used in academia include μ-FT-IR (micro-Fourier Transform Infrared) spectroscopy using a Fourier transform infrared spectrophotometer and μ-Raman spectroscopy using a Raman spectrometer.

In spectroscopic analysis, the mapping area and the loss rate can vary depending on the particle size of the sample, potentially leading to underestimation. Therefore, selecting appropriate instruments based on the cut-off size of the sample is critical to improving analytical accuracy. Specifically, μ-Raman is suitable for analyzing microplastics in the 5–20 μm range, while μ-FT-IR can be used for microplastics larger than 20 μm.

2.1. μ-Raman Spectrometer

Raman spectroscopy is a spectroscopic technique based on the Raman scattering phenomenon, used to analyze molecular structures and chemical compositions. This effect, discovered in 1928 by Indian physicist C.V. Raman (Chandrasekhara Venkata Raman), occurs when light of a specific wavelength is incident on a substance and a small portion of the scattered light has a different wavelength than the incident light. This provides information about vibrational, rotational, and other low-frequency modes of the molecules. Raman analysis has the advantage of being non-destructive and applicable to solids, liquids, and gases.

Figure 2: μ-Raman Spectrometer

2.2. μ-Raman Spectroscopy Procedure

A typical Raman analysis procedure is as follows:

First, atmospheric samples undergo pretreatment sufficient for Raman measurement, after which the foreign particles are filtered onto a silicon filter. Once pretreatment is complete, the Raman microscope divides the total sample area according to the camera's field of view (FOV). The XY stage of the microscope moves to each divided section, and the Z stage adjusts to achieve optimal focus, allowing an optical image of the section to be acquired.

Next, the extracted coordinates (X, Y, Z) of the Raman measurement targets are used to position the XY and Z stages for spectroscopic measurement. The Raman spectra are collected and analyzed to identify the type of particles present.

After measuring all target coordinates within a section, the system moves to the next divided section, repeating the process until all areas have been analyzed. Finally, the locations, types, and physical characteristics of the detected microplastic (MP) particles are overlaid on the optical image and summarized in tabular form.

Thus, μ-Raman-based microplastic analysis proceeds through particle localization and spectral acquisition. After completing this sequence of steps, the types of microplastics are identified through comparative analysis of the spectral features associated with each particle.

Recent advancements in imaging technology now enable recognition of different particle types and the creation of type-specific distribution maps. These developments facilitate the classification of microplastic particles by size and material type.

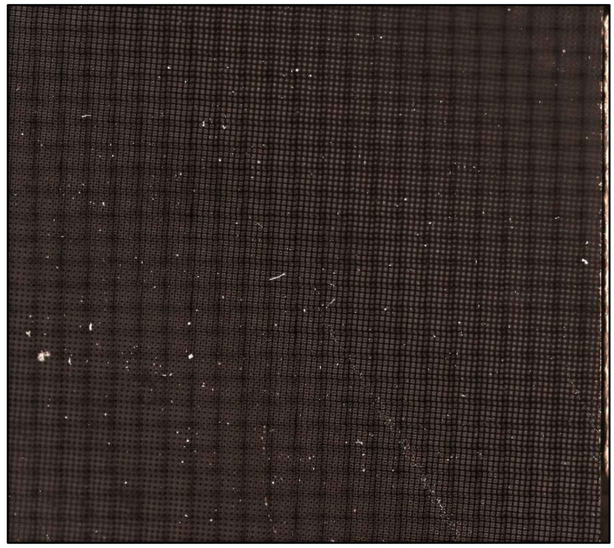

Figure 3: Filter image after mapping with μ-Raman

3. Research Methods

In this study, we aim to establish a foundation for identifying the optimal pretreatment method that ensures the highest recovery rate of microplastics prior to spectroscopic analysis using μ-Raman. This is achieved by evaluating various combinations of pretreatment techniques.

To derive the optimized method, the study proposes the following sequential pretreatment steps: **re-suspension** to detach microplastics from actual environmental samples; **oxidative treatment**, which removes various organic substances in the sample by adding 20 mL each of H_2O_2 and Fenton's reagent and maintaining the mixture at 60°C for 2 hours; and **density separation**, where detached microplastics are treated with a $ZnBr_2$ solution, allowed to settle in a separatory funnel for 24 hours, and then filtered for analysis.

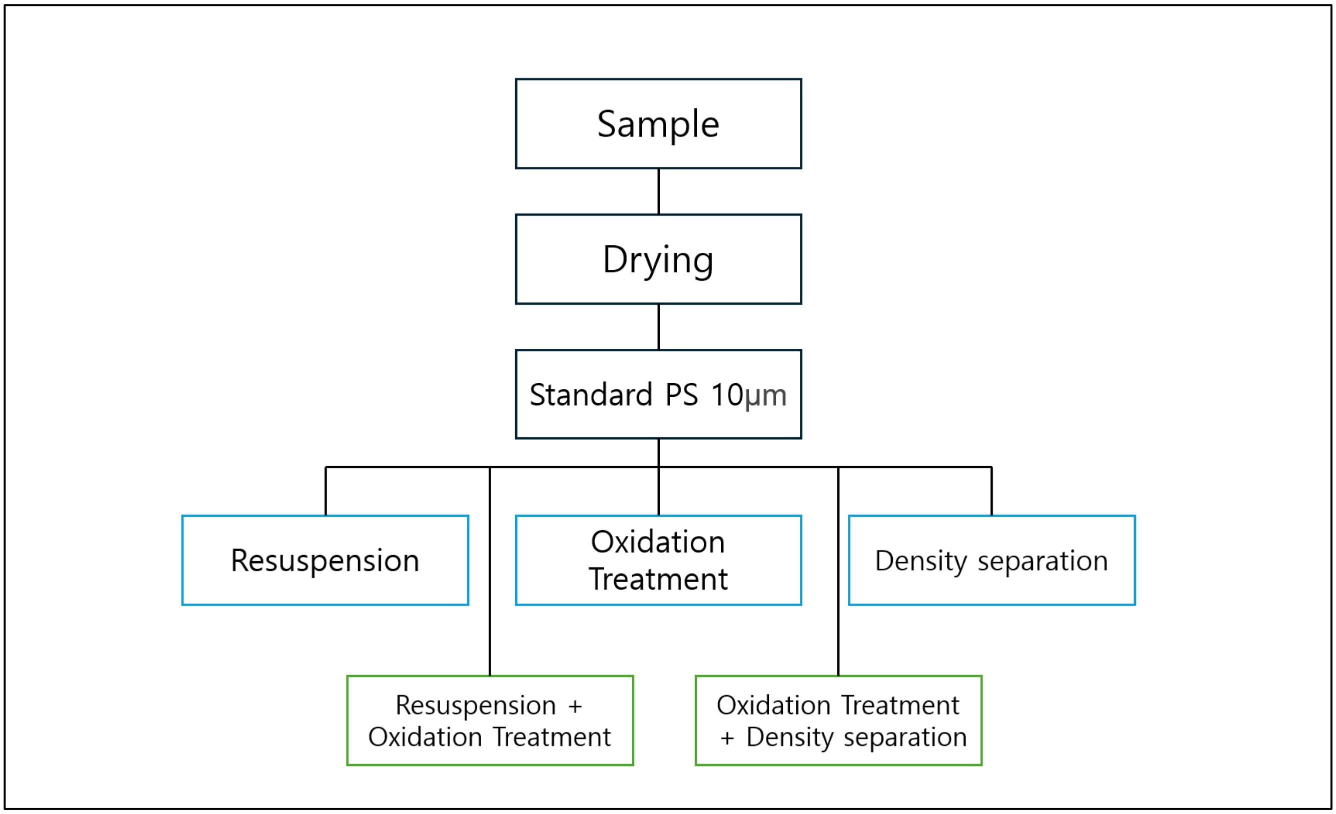

Figure 4: Workflow for Microplastic Recovery Assessment Using μ-Raman

3.1. Re-suspension Treatment

The environmental sample is subjected to sonication for 10 minutes using an ultrasonic bath. This re-suspension process helps detach microplastics adhered to the filter that collected the sample.

3.2. Density Separation

A 20 mL aliquot of $ZnBr_2$ density separation solution is added, and the actual filter sample undergoes 10 minutes of ultrasonic treatment to dislodge microplastics attached to the filter surface. The sonicated sample is then left undisturbed in a separatory funnel for 24 hours. After settling, the density separation solution is carefully decanted to prevent disturbance of the precipitate, and the supernatant is collected. The supernatant is filtered through a 5 μm SUS filter and stored in a Petri dish. If a salt precipitate forms due to the reaction of $ZnBr_2$ with H_2O_2 or distilled water, a diluted sulfuric acid solution is prepared and used during filtration to prevent interference.

3.3. Oxidative Treatment

The filter sample remaining after density separation is transferred into a cleaned container. Then, 20 mL each of H_2O_2 and Fe(II) solution are added, and the mixture is heated at 70°C for 2 hours. One hour into the heating process, an additional 20 mL each of H_2O_2 and Fe(II) solution are added. After the 2-hour oxidation is complete, multi-stage filtration is carried out using SUS filters with pore sizes ranging from 20 μm to 5 μm. This step helps eliminate larger interfering particles that could hinder the microplastic analysis.

4. Results and Discussion

Based on repeated experiments and comparisons with various existing studies on airborne microplastics, it was found that Raman spectroscopy exhibited similar trends to FT-IR spectroscopy. Moreover, quality control during the pretreatment process contributed to improving the recovery rate.

First, in the case of **re-suspension without any pretreatment**, it was observed that analysis using the μ-Raman spectrometer was not feasible due to interference from substances such as gnats, seeds, and other contaminants.

Second, when **density separation** was applied during pretreatment, the sample included a physical process (re-suspension) that helped detach microplastics. This allowed for relatively low-background analysis. Inorganic materials were precipitated and removed using a separatory funnel, indicating that interfering substances were successfully eliminated.

Third, when **oxidative treatment** was included in the pretreatment process, organic matter was chemically removed. This enabled the acquisition of relatively clearer filter images during Raman mapping compared to other pretreatment methods.

Therefore, it was concluded that **oxidative treatment should be prioritized** in the pretreatment process when analyzing actual environmental samples. This step effectively removes interfering substances and ensures that μ-Raman spectroscopic analysis can proceed without difficulty.

5. Conclusions

Through the comparative evaluation of pretreatment methods for microplastic analysis using μ-Raman spectroscopy, the following conclusions were drawn.

First, **μ-Raman spectroscopic analysis cannot be conducted without appropriate pretreatment**, as the samples typically contain significant amounts of interfering substances, including both inorganic and organic matter.

Additionally, although the **re-suspension step**—which detaches plastics from filters—may result in some loss of microplastics, this step is essential when analyzing actual environmental samples, as it ensures effective detachment. Thus, re-suspension is a **necessary step** for the practical application of standardized microplastic analysis.

Considering that **density separation inherently includes re-suspension**, the following combinations of pretreatment processes can be proposed:

1. Re-suspension
2. Oxidative treatment
3. Density separation
4. Re-suspension + Oxidative treatment
5. Density separation + Oxidative treatment

By applying **density separation and oxidative treatment consecutively after re-suspension**, it is possible to remove both organic and inorganic interfering substances from the sample, thus deriving a pretreatment strategy that ensures optimal microplastic recovery.

Furthermore, to ensure **high recovery rates and reproducibility** throughout the experiment, certain **quality control measures** must be implemented:

- Due to the high susceptibility of microplastic samples to contamination, all pretreatment processes—except during reagent addition—should be performed with the sample sealed using aluminum foil or other protective covers.
- Filters should be stored in Petri dishes made of **glass or stainless steel with a fitted lid**, and the dish should be at least 5 mm larger than the filter to avoid loss.
- During all pretreatment procedures, especially when using glass funnels, care should be taken to **rinse and recover any residual microplastics** using distilled water or the previously applied reagents to maximize recovery rates.

In conclusion, if the results and analytical procedures presented in this study are **cross-validated and proven reliable**, the proposed pretreatment and analysis workflow can serve as a **standardized method for airborne microplastic analysis using μ-Raman spectroscopy**. These findings may thus contribute to establishing foundational guidelines for the standardization of atmospheric microplastic analysis protocols.

References

Carey, P. (2012). *Biochemical applications of Raman and resonance Raman spectroscopes*. Elsevier.

Czamara, K., Majzner, K., Pacia, M. Z., Kochan, K., Kaczor, A., & Baranska, E. M. (2015). Raman spectroscopy of lipids: a review. *Journal of Raman spectroscopy*, *46*(1), 4-20.

Czamara, K., Majzner, K., Pacia, M. Z., Kochan, K., Kaczor, A., & Baranska, E. M. (2015). Raman spectroscopy of lipids: a review. *Journal of Raman spectroscopy*, *46*(1), 4-20.

Dresselhaus, M. S., Dresselhaus, G., Saito, R., & Jorio, A. (2005). Raman spectroscopy of carbon nanotubes. *Physics reports*, *409*(2), 47-99.

Gardiner, D. J. (1989). Introduction to Raman scattering. In *Practical Raman Spectroscopy* (pp. 1-12). Berlin, Heidelberg: Springer Berlin Heidelberg.

Graves, P. R. G. D. J., & Gardiner, D. (1989). Practical raman spectroscopy. *Springer*, *10*, 978-3.

Hanlon, E. B., Manoharan, R., Koo, T., Shafer, K. E., Motz, J. T., Fitzmaurice, M., ... & Feld, M. S. (2000). Prospects for in vivo Raman spectroscopy. *Physics in Medicine & Biology*, *45*(2), R1.

Jones, R. R., Hooper, D. C., Zhang, L., Wolverson, D., & Valev, V. K. (2019). Raman techniques: fundamentals and frontiers. *Nanoscale research letters*, *14*, 1-34.

Keresztury, G., Chalmers, J. M., & Griffith, P. R. (2002). Raman spectroscopy: theory. *Handbook of vibrational spectroscopy*, *1*, 71-87.

Koningstein, J. A. (2012). *Introduction to the Theory of the Raman Effect*. Springer Science & Business Media.

Kudelski, A. (2008). Analytical applications of Raman spectroscopy. *Talanta*, *76*(1), 1-8.

Kudelski, A. (2009). Raman spectroscopy of surfaces. Surface Science, 603(10-12), 1328-1334.

Loudon, R. (1964). The Raman effect in crystals. Advances in Physics, 13(52), 423-482.

Loudon, R. (2001). The Raman effect in crystals. *Advances in Physics*, *50*(7), 813-864.

Lyon, L. A., Keating, C. D., Fox, A. P., Baker, B. E., He, L., Nicewarner, S. R., ... & Natan, M. J. (1998). Raman spectroscopy. *Analytical Chemistry*, *70*(12), 341-362.

Malard, L. M., Pimenta, M. A., Dresselhaus, G., & Dresselhaus, M. S. (2009). Raman spectroscopy in graphene. *Physics reports*, *473*(5-6), 51-87.

McCreery, R. L. (2005). *Raman spectroscopy for chemical analysis*. John Wiley & Sons.

Moon, J., Li, M., Ramirez-Cuesta, A. J., & Wu, Z. (2023). Raman spectroscopy. In *Springer handbook of advanced catalyst characterization* (pp. 75-110). Cham: Springer International Publishing.

Mulvaney, S. P., & Keating, C. D. (2000). Raman spectroscopy. *Analytical Chemistry*, *72*(12), 145-158.

Orlando, A., Franceschini, F., Muscas, C., Pidkova, S., Bartoli, M., Rovere, M., & Tagliaferro, A. (2021). A comprehensive review on Raman spectroscopy applications. *Chemosensors*, *9*(9), 262.

Robert, B. (2009). Resonance raman spectroscopy. *Photosynthesis research*, *101*, 147-155.

Rostron, P., Gaber, S., & Gaber, D. (2016). Raman spectroscopy, review. *laser*, *21*, 24.

Rygula, A., Majzner, K., Marzec, K. M., Kaczor, A., Pilarczyk, M., & Baranska, M. (2013). Raman spectroscopy of proteins: a review. *Journal of Raman Spectroscopy*, *44*(8), 1061-1076.

Schrader, B. (Ed.). (2008). *Infrared and Raman spectroscopy: methods and applications*. John Wiley & Sons.

Seung Jun, W. O. O. (2025). A Study on the Current Status of Microplastic Analysis in the Air Using FT-IR. *Journal of Wellbeing Management and Applied Psychology*, *8*(1), 1-5.

Shipp, D. W., Sinjab, F., & Notingher, I. (2017). Raman spectroscopy: techniques and applications in the life sciences. *Advances in Optics and Photonics*, *9*(2), 315-428.

Singh, R. (2002). CV Raman and the Discovery of the Raman Effect. *Physics in Perspective*, *4*, 399-420.

Smith, E., & Dent, G. (2019). *Modern Raman spectroscopy: a practical approach*. John Wiley & Sons.

Szymanski, H. A. (2012). *Raman spectroscopy: theory and practice*. Springer Science & Business Media.

Vandenabeele, P. (2013). *Practical Raman spectroscopy: an introduction*. John Wiley & Sons.

Won, S., Lee, I., Park, J., Park, J., & Kim, H. (2023). A Study on the Distribution Characteristics of Microplastics in the Atmosphere Using Spectroscopic Analysis. *Journal of Environmental Analysis, Health and Toxicology*, *26*(4), 181-190.