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## Pilot Study on the Removal of Microplastic Contamination from Spices Resulting from the Use of Pepper Mills with Plastic Grinding Mechanisms

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### **Abstract:**

**Purpose:** This study analyzed the possibility of using magnetic iron oxide ( $Fe_3O_4$ ) nanoparticles as sorbents to capture microplastics released during pepper grinding. The synthesized and functionalized  $Fe_3O_4$  nanoparticles were evaluated for their adsorption and separation capabilities under laboratory conditions.

**Approach/Methodology/Design:** Microplastics (MP) are increasingly being identified in food products, and one potentially underestimated source of their presence in the diet is kitchen equipment. In particular, pepper mills with plastic burrs can generate MP particles as a result of mechanical abrasion.

**Findings:** The results indicate that nanomagnetic sorbents may be a promising method for reducing exposure to microplastics from kitchen accessories. At the same time, it is necessary to further investigate the environmental and biological safety associated with the use of  $Fe_3O_4$  nanoparticles and to develop strategies for their regeneration and immobilization.

**Practical Implications:** It was demonstrated that the application of an external magnetic field enables the effective removal of MP particles from aqueous solutions, with an average capture efficiency of approximately 40% (recovery between 30–60%).

**Originality/Value:** FTIR qualitative analysis confirmed the presence of microplastics in samples of ground pepper from mills with plastic burrs.

**Keywords:** Nano-iron, hydrophobic adsorption, microplastics.

**JEL classification:** Q53, Q56, I18, L66, O13.

**Paper type:** Research article.

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## **1. Introduction**

In recent years, the issue of microplastics (MP) and nanoplastics (NP) has emerged as one of the most pressing environmental challenges. Microplastics (MP) are now being detected in a variety of food products, and one of the less obvious sources of contamination is kitchen equipment. Increasing attention is being paid to spice mills equipped with plastic grinding wheels, which can wear down during grinding and lead to the release of microparticles into food. In the case of pepper, a spice consumed daily around the world, this is a potentially significant source of exposure to MP (Chen *et al.*, 2023).

In mills with plastic grinding stones for grinding pepper, mechanical friction between the hard peppercorns and the surface of the grinding stones causes gradual degradation of the plastic used. This results in the breaking off of polymer fragments (mainly polypropylene, polyamide, or ABS), generating microplastic particles ranging in size from 10 to 500  $\mu\text{m}$ , which end up directly in food, and the accumulation of trace amounts of nanoplastics ranging in size from 1 nm to 1  $\mu\text{m}$ , which are difficult to detect using traditional methods (Liu and Zhang, 2024).

Simulation studies have shown that intensive use of mills with plastic burrs can lead to the release of several hundred to several thousand MP particles per 100 g of ground pepper (Ramos *et al.*, 2022). These amounts vary depending on the quality of the material, frequency of use, and degree of wear on the burrs. Comparative experiments indicate that mills with ceramic or steel burrs emit significantly less or no microplastics at all (Yang *et al.*, 2023).

Regular consumption of ground pepper from plastic mills may be an underestimated source of MP exposure in the diet. Potential consequences include: physical damage to the intestinal epithelium by sharp-edged plastic particles, the transfer of chemical additives (phthalates, BPA) into the body, and effects on the gut microbiota and metabolism (Yong *et al.*, 2020). Although the amounts of MP in a single dose are relatively small, long-term accumulation poses a significant health risk.

Unfortunately, there are currently no clear regulations on microplastic emissions from kitchen appliances. However, a growing number of studies suggest the need to introduce quality tests and MP emission standards for kitchen accessories, promote alternative materials (ceramics, stainless steel), and raise consumer awareness about choosing the right kitchen tools (EFSA, 2023).

In recent years, technologies based on nanomaterials, in particular magnetic iron oxide nanoparticles ( $\text{Fe}_3\text{O}_4$ ), have been intensively developed (Xu *et al.*, 2009; Liu *et al.*, 2010; Taghizadeh *et al.*, 2013; Samanta and Ravoo, 2014; Santhosh *et al.*, 2016; Qu *et al.*, 2022). Due to their adsorption properties and ease of magnetic separation, these nanoparticles are an attractive alternative to traditional adsorbents. Their unique features, such as high specific surface area, magnetic reactivity, and

reusability, enable effective capture of various pollutants, including microplastics (Tang *et al.*, 2021; Shi *et al.*, 2022; Heo *et al.*, 2022; Shen *et al.*, 2023).

The concept of nanomagnetic capture involves functionalizing the surface of Fe<sub>3</sub>O<sub>4</sub> nanoparticles so that they can bind to microplastics and then be easily separated using an external magnetic field. Fe<sub>3</sub>O<sub>4</sub> magnetic nanoparticles are characterized by a large specific surface area and a strong response to a magnetic field. After surface modification—e.g., by coating with polymers, surfactants, or functional groups—they can form complexes with microplastics in water. Subsequently, thanks to the application of a magnetic field, the MP-nanoFe<sub>3</sub>O<sub>4</sub> complexes are easily removed.

Recent innovations include: coating nanoparticles with hydrophobic ligands to increase their affinity for plastic surfaces (Zhou *et al.*, 2023), the use of multifunctional nanoparticles (e.g., Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub> with amino groups) that enable simultaneous MP capture and heavy metal removal (Liu *et al.*, 2024), and the integration of nanomagnetic sorbents into filter membranes which improves separation efficiency under real-world conditions (Chen *et al.*, 2024). Laboratory studies have shown that nanomagnetic sorbents can remove up to 90–99% of microplastic particles of various sizes from drinking water and wastewater (Wu *et al.*, 2023).

Recent research has extensively explored the adsorptive properties of nano-iron and its applications in pollutant removal (Shi *et al.*, 2022; Heo *et al.*, 2022). For instance, micron-sized polystyrene (PS) has been used as a model microplastic in adsorption studies. These studies examined the adsorption kinetics and isotherms, as well as the effects of polymer size on efficiency. Electrostatic and hydrophobic interactions were identified as primary mechanisms, and the reusability of Fe<sub>3</sub>O<sub>4</sub> via desorption was also demonstrated (Heo *et al.*, 2022).

Another study showed that the removal efficiency of four common microplastics—polyethylene (PE), polypropylene (PP), polystyrene (PS), and polyethylene terephthalate (PET)—with particle sizes between 200 and 900 µm reached up to 86.87 ± 6.92%, depending on polymer type and particle size. Removal efficiency was positively correlated with the density of Fe<sub>3</sub>O<sub>4</sub> nanoparticles on the microplastic surface (Shi *et al.*, 2022).

Additionally recent research (Shi *et al.*, 2023) investigated PS removal using biochar modified with cetyltrimethylammonium bromide. Despite electrostatic repulsion, PS showed high stability in alkaline conditions, with a 67.4% removal efficiency at pH 11, while initial increases in NaCl concentration reduced PS removal, the effect plateaued at higher concentrations.

Magnetic nanoparticles (MNPs) are of particular interest due to their unique properties—superparamagnetism, magnetoresistance, and magneto-optics—not observed in bulk materials. Their small size results in enhanced surface activity and

shape-dependent behavior (Lu *et al.*, 2007; Issa *et al.*, 2013; Binandeh, 2022). Among them, superparamagnetic iron oxide nanoparticles (SPIONs), such as magnetite ( $\text{Fe}_3\text{O}_4$ ), maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ), and doped ferrites (e.g., Co, Ni, Cu), have attracted attention due to their biocompatibility, chemical stability, and affordability (Kudr *et al.*, 2017).

However, due to their tendency to agglomerate, MNPs often require stabilization with organic or inorganic coatings. Surface functionalization—e.g., with polymers or biomolecules—helps to tailor particle properties for specific applications (Díez *et al.*, 2022). In this study, silane (HDTMS) was used to modify the surface hydrophobicity of  $\text{Fe}_3\text{O}_4$ , reducing aggregation and enhancing selective interaction with plastic particles. This surface modification also conferred multifunctionality (Zhu *et al.*, 2018; Gambhir *et al.*, 2022).

Magnetic separation is gaining recognition as an efficient and scalable method for the removal of MPs and NPs from aqueous environments. It combines cost-effectiveness, availability, and rapid separation with the advantages of high surface area and magnetic recoverability of nanoparticles (de Las Nieves Piña *et al.*, 2018; Li *et al.*, 2021).

For instance,  $\text{Fe}_3\text{O}_4$ -based particles demonstrated >80% removal efficiency for PE, PP, PS, and PET (200–900  $\mu\text{m}$ ) (Shi *et al.*, 2022). Other studies have shown that mixing  $\text{Fe}_3\text{O}_4$  nanoparticles with PS microparticles enabled their complete removal from water within one minute, even in natural river samples (Heo *et al.*, 2022). The interaction was largely attributed to hydrophobic forces, and efficiency improved with increasing adsorbent concentration.

The aim of this pilot study was to investigate the possibility of using nanoiron to capture microplastics generated during the grinding of pepper in plastic mills. The study presents an experimental design, the characteristics of the nanomaterial, and an assessment of the effectiveness of magnetic capture of microplastics, with particular emphasis on the quantitative and qualitative analysis of the recovered particles.

The best documented sources of dietary exposure for microplastics (MP) are seafood, and to a lesser extent salt, honey, bottled water, and other products. However, the data are very heterogeneous in terms of methodology (Ziani *et al.*, 2023).

What about the health effects of microplastics? According to the World Health Organization current data do not yet allow for a full quantitative assessment of health risks, but there are sufficient grounds to treat MP/NP as a potential threat. The European Commission has developed a methodology for monitoring microplastics in drinking water, and in 2025, reference materials were introduced to enable better validation of laboratory methods. EFSA points to the need to standardize toxicological methods and study the bioavailability of nanoplastics.

EFSA has been conducting horizontal work on emerging risks for several years, in 2024, it was emphasized that there is a lack of robust toxicological and exposure data for a full risk assessment of MP/NP in food (especially NP <1 µm). The priorities are: standardization of analytics, bioavailability and toxicokinetics of NP, and the impact of co-contaminants (Gkrintzali *et al.*, 2024).

Scientific evidence from recent years clearly indicates that MP/NP penetrate the human body and can affect critical biological systems. The strongest signals concern an increased risk of cardiovascular disease and possible fertility disorders. Although causality has not yet been fully confirmed, the observed correlations and mechanistic experimental data provide a strong argument for further monitoring and limiting exposure.

Key challenges for the coming years include the development of nanoplastics detection methods, cohort studies, and the assessment of toxicokinetics in humans (Ramsperger *et al.*, 2020).

Although nanomagnetic MP capture is a promising technology, questions arise about the safety of releasing Fe<sub>3</sub>O<sub>4</sub> nanoparticles into the environment. Potential effects include: toxicity to aquatic organisms – excess nano-Fe<sub>3</sub>O<sub>4</sub> can generate reactive oxygen species, causing oxidative stress in fish and invertebrates, bioaccumulation – nanoparticles can penetrate tissues and enter the food chain, impact on the aquatic microbiome – disruption of the balance of microorganisms responsible for natural biogeochemical processes (Zhang *et al.*, 2022).

Therefore, it is necessary to develop methods for the immobilization and regeneration of nanoparticles that will minimize the risk of their entry into ecosystems.

## 2. Materials and Methods

The advantage of using the magnetic microplastic (MP) extraction method, as described by Grbic and co-workers (Grbic *et al.*, 2019), lies in its low cost and high potential for further development. In this study, iron nanoparticles (nano-Fe) from Sigma-Aldrich were used. The nanoparticles were pre-treated with HDTMS silane to hydrophobize their surface and improve their interaction with plastic particles.

Nano-Fe modified with HDTMS was synthesized in methanol. The modified nanoparticles were then used to capture large plastic particles (>1 mm), medium-sized ones (200 µm to 1 mm), and small particles (<20 µm) from aqueous solutions using a neodymium magnet (N52).

The described magnetic MP capture method involved using nano-Fe with enhanced hydrophobicity through HDTMS silane modification. All reagents were sourced from Sigma-Aldrich (Merck). Due to limited availability and distribution of these

materials in Europe, a comparable substitute with similar particle size and properties was sourced from the German company Io-Li-Tec (Ionic Liquids Technologies). The method was adapted to separate MPs originating from the abrasion of disposable spice grinder burrs from ground pepper. The approach was based on a method developed by Ramage and co-workers (Ramage *et al.*, 2022), in which MPs were separated from soil using iron nanoparticles modified with HDTMS and an electromagnet.

Portions of nano-Fe purchased from Io-Li-Tec were modified with HDTMS in ethanol 2h in room temperature, evaporated, and subsequently reacted with MPs derived from plastic grinder components found in ground pepper. Pepper samples containing plastic were introduced into an aqueous solution, and a neodymium magnet N52 (Magnepol) was placed inside a glass cylinder to extract the MPs. The material was then dried on pre-weighed filter paper, and the mass of retained MPs bound to nano-Fe was determined by differential weighing.

Five types of pepper grinders with polymer burrs—types 1(A), 2(B), 3(C), 4(D), and 5(E)—were purchased from grocery stores in Poland.

Fourier-transform infrared spectroscopy (FTIR) is a widely used technique for identifying organic materials and has recently gained popularity for characterizing the chemical composition of unknown plastic fragments (Veerasingam *et al.*, 2021).

The MPs bound to nano-Fe and derived from grinder burrs were analyzed using an AIM-9000 (Automatic Infrared Microscope) combined with an IRTracer-100 spectrophotometer (Shimadzu). Thanks to its highly sensitive TGS detector, reflection analysis of the tested materials was possible. These spectra were compared with original polymer samples collected from the grinder burrs and matched with polymer reference spectra from the Shimadzu library (Zbucki and Plažuk, 2024).

Additionally, for comparison, MPs were quantified after digestion of ground pepper using a NaClO/KOH (1:1) solution, according to the method by Strand and Tairova (Strand & Tairova, 2015), to eliminate organic matrix interference. The resulting solution was filtered using syringes and G8 glass fiber filters (Fisher Scientific). The recovered polymer particles were counted using a bacterial colony counter with a magnifying chamber and lens (30-CH-20 Alfa Chem), under 3× magnification and with five replicates.

For quantification, a blind control (only nano-Fe + pepper) was used to prove that the weight increase was solely due to MPs. The results also included the weight of HDTMS to rule out matrix interference. Filters were stored in sealed glass Petri dishes to avoid contamination from air (to exclude environmental background) and were analyzed using the FT-IR IRT-100 / AIM-9000 system (Shimadzu). The major polymers used in grinder components were identified using Shimadzu's polymer spectral database.

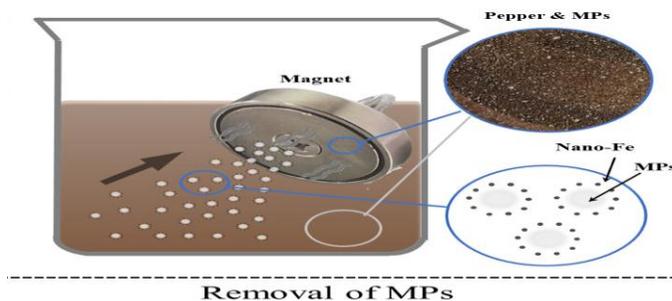
Magnetic adsorbents are widely used to remove common pollutants such as organic contaminants and heavy metal ions. More importantly, magnetic nanoparticles have shown significant potential in the adsorption-based removal of widespread NP/MP particles—something traditional adsorbents often fail to achieve (Shi *et al.*, 2022). The adsorption efficiency of these contaminants can likely be improved through the use of rationally designed magnetic adsorbents based on established adsorption mechanisms.

In this pilot study, magnetic capture of microplastic fragments from ground pepper was proposed as a method that could also be useful for removing plastic particles from both environmental samples and food products. The strategy involved the use of  $\text{Fe}_3\text{O}_4$ , and the study identified certain limitations of magnetic adsorbents in practical applications. Experimental Design and Procedure for Magnetic Capture of Nano-Iron with Adsorbed MPs (Figure 1):

- Experimental design and adaptation to the research model;
- Preparation and verification of nano-Fe hydrophobized with HDTMS silane;
- Testing magnetization and capture of known MP particles;
- Performing the experiment with 5 repetitions for each of the 5 commercially available disposable pepper grinders;
- Differential mass determination of MPs based on weighing filter paper with magnetically captured MPs bound to nano-Fe (considering the mass of nano-Fe and HDTMS accompanying MPs on each filter);
- FTIR identification of polymers and comparison with previously obtained spectra from MPs abraded directly from grinder components;
- Comparison of results with polymer spectra from the Shimadzu FTIR reference library.

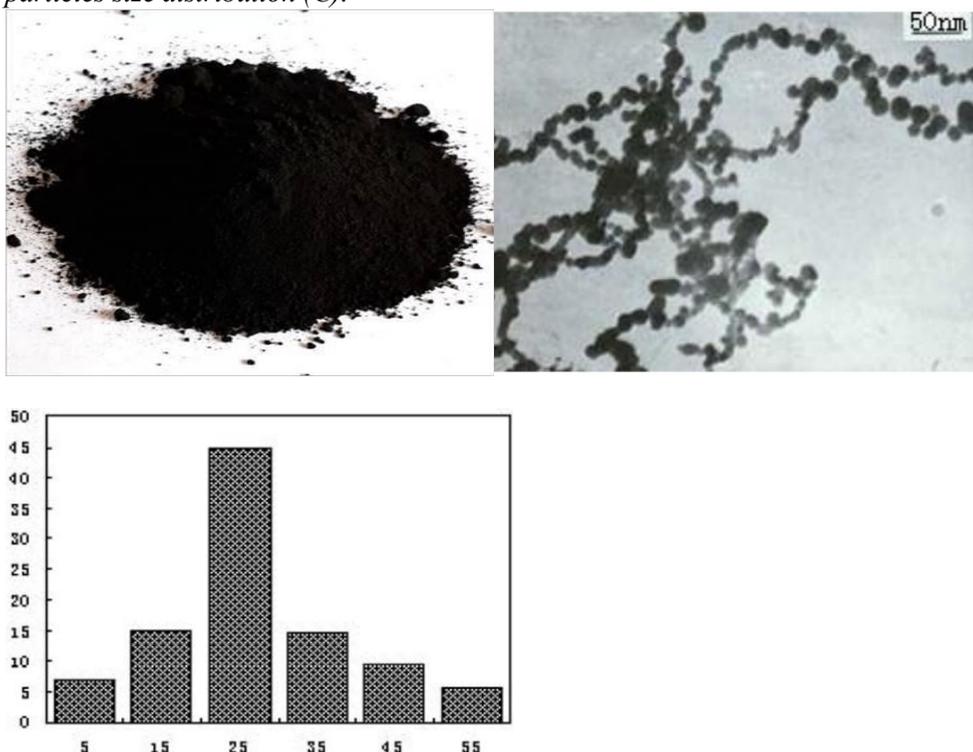
The nano-iron used in the experiments was characterized as having a particle size of approximately 25 nm, as confirmed by transmission electron microscopy (Figure 2 A, B, C).

**Figure 1.** Schematic representation of the research assumptions - microplastic removal by magnetic capture using nano-Fe



**Source:** <https://www.sciencedirect.com/science/article/abs/pii/S0048969721049135>.

**Figure 2.** Nanoiron specification: powder formulation (A), TEM images (B) and particles size distribution (C).



*Source: Own study.*

### 3. Results and Discussion

As a result of the conducted experiment, using FTIR spectroscopy and the Shimadzu reference library, the primary polymers comprising the abrasion-prone components of each grinder type were identified as follows:

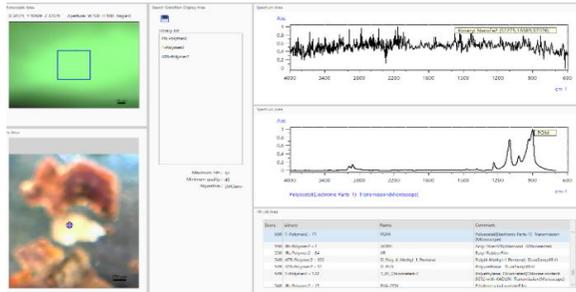
A – POM, B – POM, C – Nylon, D – PMMA, and E – PC (Figure 3 A–E). These findings were subsequently confirmed via magnetic capture using the AIM-9000 infrared microscope (Figure 3 A–E) and were consistent with previous analyses (Zbucki and Plažuk, 2024).

As shown in Table 1, preliminary estimations suggest that the amount of MPs generated through the abrasion and wear of plastic components in pepper grinders may be below 0.1% relative to the total content of spices in such grinders. The magnetic capture efficiency varied between grinder types, ranging from  $29.96 \pm 12\%$  to  $69.56 \pm 13.67\%$ , with the highest efficiency observed in grinder type C, where the primary polymer component of the burrs was nylon. Grinders made predominantly

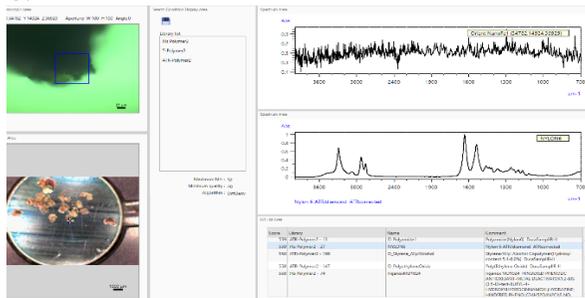


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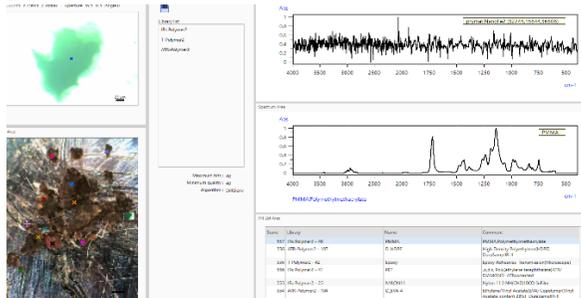
**B.**



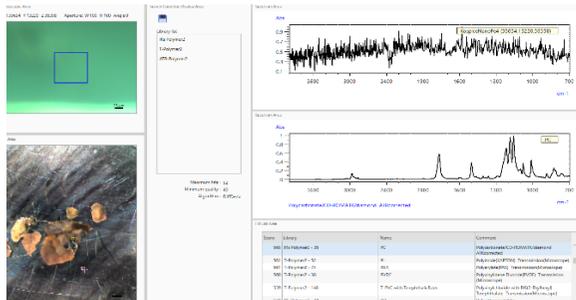
**C.**



**D.**



**E.**



Source: Own study.

The results of this study confirm that iron oxide nanoparticles ( $\text{Fe}_3\text{O}_4$ ) are effective magnetic adsorbents for capturing microplastic particles generated by abrasion of plastic components in pepper grinders. The observed adsorption efficiency highlights the potential of magnetic nanomaterials for removing micro- and nanoplastic contaminants, an application of growing importance in the context of environmental protection and public health.

Advantages of this approach include:

- I. **High efficiency:**  $\text{Fe}_3\text{O}_4$  nanoparticles can effectively capture microplastics, including those of very small size.
- II. **Ease of separation:** Their magnetic properties enable simple and effective separation from aqueous media.
- III. **Low environmental risk:** Only small quantities of nano-iron are needed, and they can be readily removed after application, minimizing potential environmental toxicity.
- IV. **Scalability:** The method is adaptable across scales—from small-scale water purification to large-scale industrial applications.

However, several challenges and limitations must be addressed:

**Durability and stability:** Controlling particle size and ensuring long-term environmental stability is essential to prevent secondary contamination.

**Cost:** Although current production methods for nano-iron are expensive, ongoing technological advancements and economies of scale may improve cost-effectiveness.

**Ecological risk:** While  $\text{Fe}_3\text{O}_4$  nanoparticles are considered low in toxicity, their accumulation in aquatic environments could have unintended ecological consequences, necessitating close monitoring.

Previous our studies have demonstrated significant wear of plastic grinding components in all tested spice grinders (Zbucki and Plażuk, 2024), confirmed by scanning electron microscopy (SEM) and weight comparisons across different stages of use. Consistent with those findings, the highest degree of wear was observed in grinder type C, which contains as a main polymer black nylon. This was further research through FTIR and comparative analyses throughout this study (Zbucki and Plażuk, 2024).

Although many studies have reported the widespread presence of microplastics in food products, efforts to quantify plastic contamination in food remain in their infancy. Key challenges include methodological limitations and a lack of comprehensive toxicological data on microplastic exposure.

Current techniques for detecting and characterizing microplastic particles are often insufficient in terms of precision, time efficiency, or cost-effectiveness.

Terminological inconsistencies, particularly regarding micro- and nanoplastics, and analytical difficulties in quantifying these particles during food processing without degrading them further complicate reliable assessment.

This pilot study contributes to the development of reproducible procedures for isolating microplastic particles from complex food matrices—in particular, ground pepper—using a simple neodymium magnet and hydrophobic-modified Fe<sub>3</sub>O<sub>4</sub> nanoparticles.

After magnetic separation, the isolated plastic particles should undergo chemical and visual characterization. However, current methods still encounter difficulties in reliably identifying and quantifying low-mass MP and NP particles in particular, due to their weak signals in optical, spectroscopic, and spectrometric analyses. Trade-offs between throughput, spatial resolution, and analytical sensitivity persist.

A key question arising from the method evaluated here is whether the magnetic capture of particles is limited to microplastics released from the grinder during use, or whether it also encompasses other contaminants introduced during pepper cultivation, processing, or packaging. It is likely that both primary (from the device) and secondary (from external sources) contaminants are involved.

Our results suggest that magnetic separation using silane-modified iron nanoparticles is a promising, inexpensive, and accessible method for capturing and quantifying microplastic particles. Furthermore, FTIR analysis supports the chemical identification of isolated contaminants, highlighting the potential of this approach for use in food safety assessments and environmental monitoring.

In further research, it will be important to expand the mechanism of action, characterize the interactions between molecules, and link the results of this pilot test to practical applications: can the methodology be adapted for home use, how can it be scaled up for industrial processing?

Despite these promising developments, further research is required before nanoparticle-based methods for MP/NP removal can be scaled to industrial levels. Iron oxide nanoparticles—due to their environmental safety and cost-effectiveness—are strong candidates for future application.

However, optimization of nanoparticle size, coating, and synthesis remains essential for maximizing removal performance.

In the future, the development of nanomagnetic technologies will move towards: standardization of methods for testing efficacy and safety, design of biocompatible coatings to prevent toxicity, integration of the technology into industrial-scale water treatment systems, and research on the life cycle of nanomaterials to assess their long-term effects.

#### 4. Conclusions

Pepper mills with plastic burrs are an underappreciated source of microplastic contamination in food. Although their impact on total dietary exposure requires further study, there are strong indications that choosing ceramic or steel mills is a safer option. Further research and regulatory action are needed to reduce this hidden but widespread channel of MP exposure.

Nanomagnetic MP capture using iron nanoparticles is an innovative and effective method for removing plastic pollution from aqueous solutions. This technology offers high practical potential, but requires further research on environmental and health safety. It will be crucial to develop regulations and standards to ensure its responsible implementation.

**Authors' participation:** *L.Z. - conceptualization, funding acquisition, project administration, supervision; E.P. - methodology, resources, investigation, writing – original draft, M.Z-L. - formal analysis, writing – review & editing.*

**Declaration of competing interest:** *Authors declare that there is no conflict of interest. The authors declare that they have no known competing interests that are relevant to the content of this article.*

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