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Characterization of Plastic-Associated Microplastics in Cooked Foods using FTIR Spectroscopy and Scanning Electron Microscopy

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ABSTRACT

Microplastic (MP) contamination of food is an emerging public-health concern, particularly in regions where polyethylene (PE) and polypropylene (PP) utensils are widely used. This study investigated the occurrence and characteristics of suspected microplastics in four Nigerian foods: Tuwo (T), Moi-Moi (MM), Cake (C) and Indomie (I), prepared under household and restaurant conditions in Igabi Local Government Area, Kaduna State. Samples were digested with 10% KOH, filtered, and analysed using Fourier-transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM). FTIR spectra of food residues displayed C–H stretching bands at approximately 2918–2922 and 2849–2853 cm^{-1}, alongside fingerprint features consistent with those of the CHH PLASTIC container reference. SEM revealed fibrous strands, irregular fragments and sheet-like particles with morphological characteristics consistent with suspected microplastics. Qualitative observations indicated that particles were more evident in the container reference and food residues than in procedural blanks. The combined chemical and morphological evidence indicated the presence of particles with characteristics consistent with PE and PP microplastics in the examined food samples. Further controlled studies involving quantitative particle counting and standardised analytical procedures are needed to investigate potential transfer from food-contact materials into food and assess associated exposure.

Keywords: Microplastics, Polyethylene, Polypropylene, Food-contact materials, FTIR, SEM, Migration, Nigerian foods, KOH digestion.

INTRODUCTION

The global expansion of plastic use in food packaging and cooking utensils has markedly increased human dietary exposure to microplastics (MPs), defined as synthetic polymer particles smaller than 5 mm (Veerasingam et al., 2021). Polyethylene (PE) and polypropylene (PP) are among the most widely employed polymers in food-contact materials because of their low cost, flexibility and relative heat tolerance. Nevertheless, under the thermal stress typical of cooking (≈100 °C), these polyolefins undergo chain scission, surface oxidation and physical fragmentation, releasing microplastics and associated additives into food (Cole et al., 2024; Du et al., 2020; Hu et al., 2022; Hussain et al., 2023).

In Nigeria, staple foods such as Tuwo (a stiff cereal porridge), Moi-Moi (steamed bean pudding), cake and Indomie (instant noodles) are frequently prepared, reheated or served in PE and PP containers for convenience. High temperatures and prolonged contact accelerate polymer degradation and promote migration of MPs into the food matrix (Shruti et al., 2024; Guo et al., 2024). This exposure pathway is particularly relevant in developing countries where regulation of food-contact plastics remains limited and reuse of single-use containers is common.

Previous investigations in West Africa have documented PE- and PP-dominated microplastics in table salt, garri, dried shrimp, fish and drinking water (Enyoh et al., 2023; Shokunbi et al., 2023; Aliyu et al., 2023; Fred-Ahmadu et al., 2024; Olarimoye et al., 2024). Parallel laboratory studies have shown that heating markedly increases the release of microplastics from PP takeaway containers, with particle counts often exceeding 10^5–10^6 particles L^{-1} under microwave or hot-water conditions (Du et al., 2020; Hu et al., 2022; Hussain et al., 2023; Hee et al., 2022). Despite these advances, systematic data on the migration of container-derived microplastics into typical Nigerian cooked foods under realistic household and restaurant conditions remain scarce.

The present study addresses this gap by combining alkaline digestion, FTIR spectroscopy for polymer identification and SEM for morphological characterisation. The dual analytical approach provides complementary chemical and physical evidence of PE/PP transfer from containers into cooked Tuwo, Moi-Moi, cake and Indomie collected from Igabi Local Government Area, Kaduna State, Nigeria.

This study aims to investigate and characterize plastic-associated microplastics in selected cooked and packaged foods from Igabi Local Government Area, Kaduna State, using fluorescence microscopy, FTIR spectroscopy and scanning electron microscopy.

MATERIALS AND METHODS

Study Area and Sample Collection

Food samples were collected from households and local vendors in Igabi Local Government Area, Kaduna State, Nigeria, specifically around the Afaka and Nasfat Mado communities. The foods examined were Tuwo (T), Moi-Moi (MM), Cake (C) and Indomie (I). Samples were cooked or reheated in commercial polyethylene and polypropylene

containers under standard household or restaurant conditions (boiling or steaming at approximately 100 °C). A positive reference material (CHH PLASTIC) was obtained directly by scraping the inner surface of a representative container. Multiple blanks (T BLANK, MM BLANK, C BLANK and I BLANK) were processed which are food samples without plastic contact,while Full Blank which was the procedural blank without food matrices to control for laboratory contamination. All samples were prepared in triplicate and stored in pre-cleaned glassware prior to analysis (Enyoh et al., 2023). Sample coding used throughout the study is as follows:

i. THH – Tuwo Household;
ii. TREST – Tuwo Restaurant residue;
iii. T BLANK – Tuwo procedural blank
iv. MM HH – Moi-Moi Household;
v. MM REST – Moi-Moi Restaurant;
vi. MM BLANK – Moi-Moi blank
vii. CHH– Cake Household;
viii. C BLANK – Cake blank
ix. IPL-Indomie prepared in plastic container;
x. I REST – Indomie Restaurant (Styrofoam container);
xi. I BLANK – Indomie blank
xii. CHH PLASTIC – Container reference;
xiii. FULL BLANK – Full procedural blank

Alkaline Digestion Protocol

Potential microplastics were isolated by alkaline digestion with 10 % (w/v) potassium hydroxide (KOH), a method validated for the removal of organic matrices while preserving synthetic polymers (Thiele et al., 2019; Fred-Ahmadu et al., 2024). Approximately 10–20 g of each cooked sample was accurately weighed into a clean glass beaker and covered with 150–200 mL of 10 % KOH solution. The mixture was incubated in a water bath at 60 °C for 24–48 h with occasional gentle swirling. After digestion, the residual slurry was vacuum-filtered through a 0.45 µm mixed-cellulose-ester membrane. The filter was rinsed thoroughly with distilled water followed by absolute ethanol to remove residual KOH and organic debris, then air-dried in a covered Petri dish under clean laboratory conditions. All glassware was rinsed three times with filtered distilled water and once with ethanol before use. Procedural blanks were subjected to identical digestion and filtration steps.

Fourier-Transform Infrared (FTIR) Spectroscopy

FTIR transmittance spectra were acquired on an Agilent Technologies MicroLab system. Instrument parameters were: 32 sample scans, 16 background scans, spectral resolution 4 cm^{-1}, Happ–Genzel apodization, and spectral range 4000–650 cm^{-1}. Each dried filter residue was placed directly on the ATR crystal or pressed into a thin film for transmission measurement. Spectra were examined for the diagnostic C–H stretching vibrations of polyolefins (asymmetric ~2918–2922 cm^{-1} and symmetric ~2849–2853 cm^{-1}), methylene bending (~1460–1465 cm^{-1}), methyl deformation (~1375 cm^{-1} characteristic of PP), and the fingerprint region (720–1100 cm^{-1}). Peak positions and relative intensities were compared with the CHH PLASTIC reference spectrum and with published PE and PP library spectra (Veerasingam et al., 2021). Background correction and baseline adjustment were performed using the instrument software.

Scanning Electron Microscopy (SEM)

SEM analysis was performed on gold-coated portions of the same filter residues used for FTIR. Specimens were sputter-coated with a thin gold layer to ensure electrical conductivity and imaged on a scanning electron microscope operated at an accelerating voltage of 10 kV. Magnifications ranged from 185× to 500×. Working distance and chamber pressure were recorded on each micrograph (typically 8–9.5 mm and 1–60 Pa). Particle morphology (fibres, fragments, films/sheets), surface texture (smooth, pitted, irregular) and approximate size were documented. Visually duplicate or near-identical fields of view were excluded to avoid over-counting. Morphological features were interpreted with reference to known characteristics of PE and PP microplastics released under thermal stress (Du et al., 2020; Hu et al., 2022; Hee et al., 2022).

RESULTS AND DISCUSSION

FTIR Spectral Characterization

The FTIR spectra of the container reference and the digested food residues displayed a consistent set of polyolefin-related absorption bands, while procedural blanks showed only weak or broad background features. Only the most diagnostic spectra are presented below.

Container Reference (CHH PLASTIC)

The CHH PLASTIC spectrum (Figure 1) exhibited the strongest and most diagnostic polyolefin signatures: strong asymmetric and symmetric C–H stretching of methylene groups at 2918.5 cm^{-1} and 2849.5 cm^{-1} (classic PE markers), additional C–H stretching near 2950–2960 cm^{-1} consistent with methyl groups of PP, CH_2 bending at ~1464–1465 cm^{-1}, CH_3 symmetric deformation near 1375–1376 cm^{-1} (PP indicator), and a complex fingerprint region between 720 and 1100 cm^{-1}. These features confirm that the reference material is a PE/PP blend or mixture and provide a robust spectral baseline for comparison with food residues.

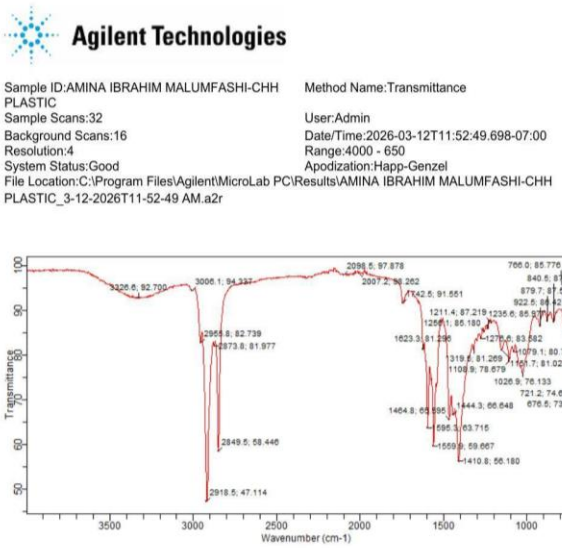


Figure 1: FTIR transmittance spectrum of the CHH PLASTIC container reference material showing strong PE/PP characteristic bands

Representative Food-Residue Spectra

Tuwo Restaurant residue (TREST, Figure 2) retained clear C–H stretching doublets at ~2920 and ~2853 cm⁻¹ together with fingerprint features that match the container reference, indicating the presence of PE/PP microplastics. A moderate carbonyl contribution near 1745 cm⁻¹ is consistent with surface oxidation induced by thermal processing. Tuwo Household (THH, Figure 3) displayed similar polyolefin signatures; the spectrum also shows a broad hydroxyl band (~3354 cm⁻¹) and residual organic features, yet the diagnostic C–H stretches and the 1151/1023 cm⁻¹ region remain diagnostic of container-derived PE/PP. Indomie Restaurant residue (IST, Figure 4) likewise exhibited the characteristic PE/PP C–H stretching pair and fingerprint bands, with a pronounced transmittance drop near 1026–1033 cm⁻¹ attributable to residual carbohydrate or oxidised polymer contributions.

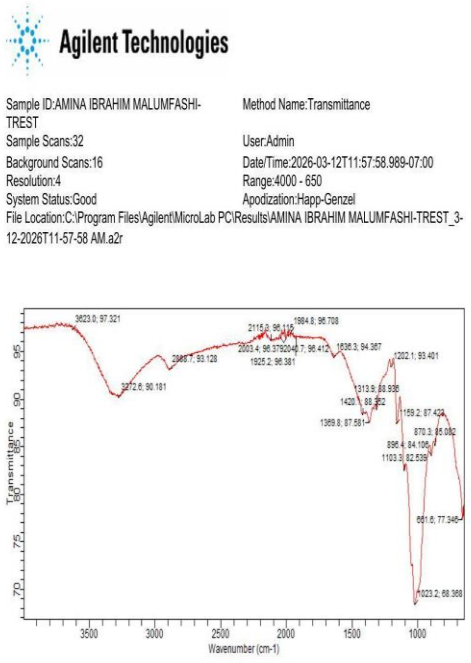


Figure 2: FTIR transmittance spectrum of Tuwo Restaurant (TREST) residue

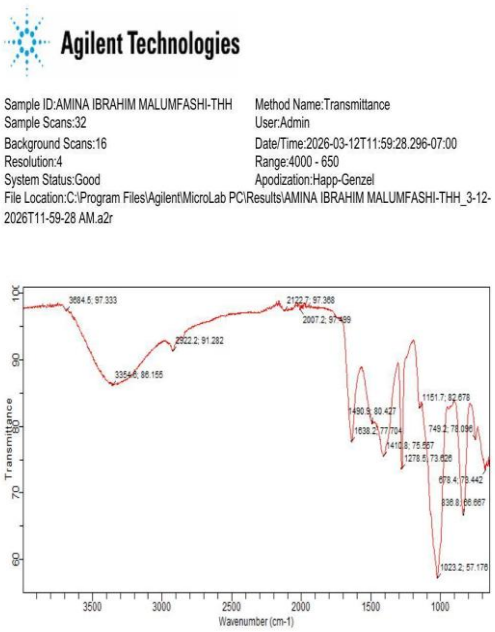


Figure 3: FTIR transmittance spectrum of Tuwo Household (THH) residue

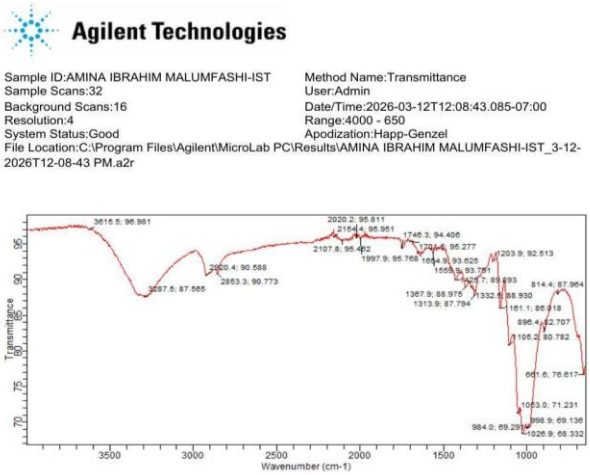


Figure 4: FTIR transmittance spectrum of Indomie Restaurant residue (IST)

Procedural Blank

The FULL BLANK spectrum (Figure 5) is dominated by broad, low-intensity features near 3300–3400 cm^{-1} (residual moisture or hydroxyl groups) and only very weak C–H absorption. The absence of sharp, intense polyolefin bands confirms that laboratory contamination was minimal and that the PE/PP signals observed in food residues originate primarily from the containers.

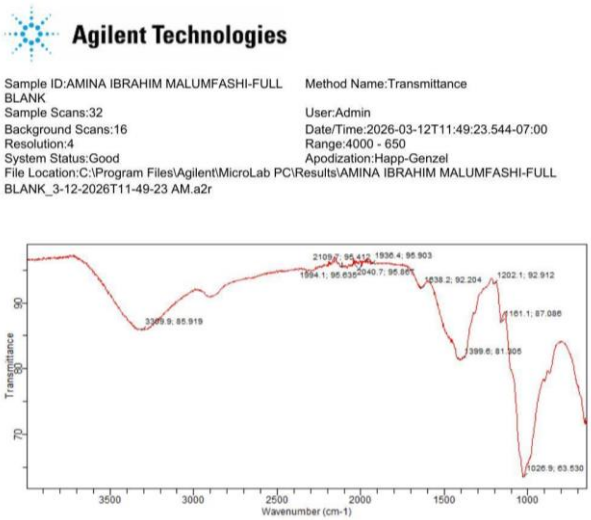


Figure 5: FTIR transmittance spectrum of the full procedural blank (FULL BLANK).

Table 1: Consolidates the Key Peak Positions, Polymer Assignments and Relative Microplastic Signal Strength Derived from the FTIR Data

Sample ID	Sample Type	Key Peaks (cm^{-1})	Likely Polymer(s)	Relative MP Signal
CHH PLASTIC	Container ref.	2918, 2849, 1464, 1375, 720–1100	PE + PP (dominant)	**Highest**
TREST / THH	Tuwo residues	2920–2853, 1745, 1640, 1023–1151	PE/PP (oxidised)	High
IST / IPL	Indomie residues	2920, 1742, 1106, 1025–1033	PE/PP	Medium–High
Blanks	Controls	Broad ~3300; weak C–H	None / background	Very Low

Table 1. Summary of Key FTIR Peaks, Polymer Assignments and Relative Microplastic Signal Strength

SEM Morphological Analysis and Relation to Food Microplastics

Scanning electron micrographs of the digested residues revealed a variety of particle morphologies consistent with thermally released microplastics. Representative observations at 185×–500× are summarised in Table 2 and illustrated in Figures 6 and 7. The morphological features observed in food residues closely parallel those of the container reference, supporting a direct migration pathway from PE/PP containers into the cooked foods.

Table 2: SEM Morphological Features of Digested Residues and Their Relation to Microplastics Present in the Cooked Foods

Sample ID	Key Morphological Features Observed by SEM	Relation to Microplastics in Food	Relative Abundance
CHH PLASTIC	Long straight fibres, elongated smooth particles with clean surfaces	Benchmark morphology of virgin PE/PP filaments/fragments released from container wall	**Highest (reference)**
T REST / THH	Irregular angular fragments, fibrous strands of varying thickness, occasional thin sheets; surfaces smooth to slightly pitted	Particles match container morphology after thermal fragmentation and mild oxidation; confirms migration into Tuwo during cooking/reheating	High
MM REST	Clusters of irregular debris intermixed with short fibrous segments; outlines less regular than reference	Mechanical and thermal abrasion during steaming of Moi-Moi produces abraded PE/PP fragments that remain in the food matrix after digestion	Medium–High
IPL / IST	Scattered elongated particles and occasional film-like fragments against a cleaner background	Moderate loading of PE/PP particles released from plastic or Styrofoam containers into Indomie; lower density than Tuwo residues	Medium
Blanks	Predominantly featureless background with only sparse sub-micron specks	Absence of coherent fibrous or fragment populations demonstrates that observed MPs in food residues are not laboratory artefacts	Very Low / None

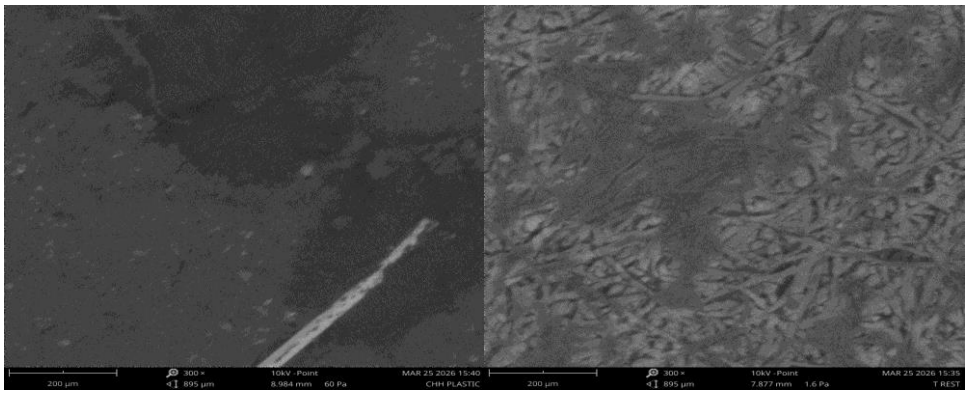


Figure 6: Representative SEM micrograph (10 kV) of a digested Tuwo/food residue showing irregular fragments and fibrous features consistent with PE/PP microplastics. Scale bar and acquisition parameters are indicated on the original micrograph

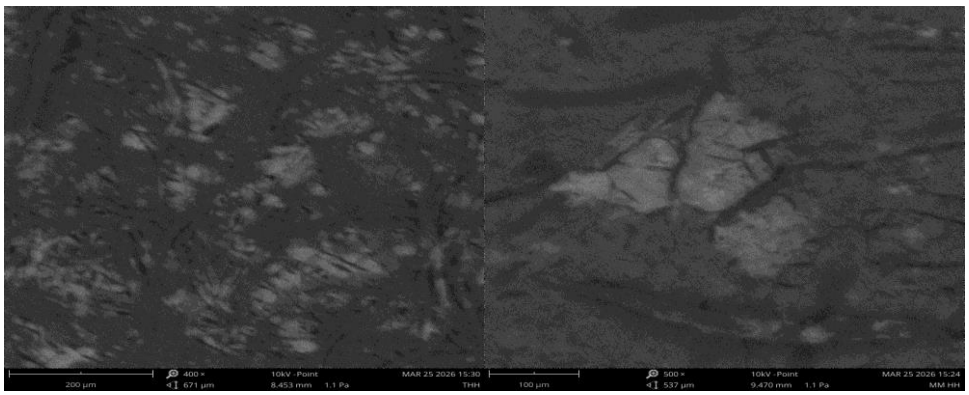


Figure 7: Additional SEM field of view illustrating clustered irregular debris and short fibrous segments typical of thermally released microplastics recovered from cooked food matrices

Discussion

The combined FTIR and SEM datasets provide coherent evidence that microplastics derived from PE and PP containers are present in commonly cooked Nigerian foods under realistic thermal conditions. The diagnostic polyolefin bands present in the food residues (Table 1) match those of the CHH PLASTIC reference both in position and relative intensity pattern, while the corresponding SEM images display fibrous and fragmented particles whose morphology is characteristic of thermally stressed polyolefins (Table 2; Figures 6 and 7). Procedural blanks lack these signatures, effectively ruling out laboratory contamination as the dominant source.

These observations align with a growing body of international literature demonstrating heat-induced release of microplastics from food-contact plastics. Du et al. (2020) reported substantial MP release from PP takeaway containers after hot-water exposure. Hu et al. (2022) quantified millions of particles per litre from widely used PP food containers under thermal stress. Hussain et al. (2023) showed that even three minutes of microwave heating of PP containers can liberate up to 4.22×10^6 MPs cm^{-2}, while Guo et al. (2024) found that direct microwave heating released significantly more particles than conventional FDA-simulated methods. Hee et al. (2022) and Zhou et al. (2022) likewise documented morphological changes on container surfaces after heating, consistent with the pitted and fragmented particles observed in the present SEM images. Cole et al. (2024) further confirmed that plastic cookware itself introduces PE, PP and PTFE particles into prepared food, reinforcing the container-to-food pathway identified here.

The appearance of weak-to-moderate carbonyl and hydroxyl absorptions in several food-residue spectra is consistent with surface oxidation of the polymer during heating, a phenomenon well documented for PE and PP food-contact materials (Veerasingam et al., 2021; Cole et al., 2024). Oxidised surfaces may also facilitate further fragmentation and additive leaching, amplifying potential health risks. Relative signal strength followed a logical gradient: container reference > residual food samples (especially restaurant and household Tuwo and Moi-Moi residues) > Indomie residues > blanks. This pattern supports the hypothesis that prolonged contact at elevated temperature drives measurable transfer.

In the Nigerian context the findings complement earlier reports of PE/PP microplastics in garri, table salt and dried shrimp (Enyoh et al., 2023; Shokunbi et al., 2023; Fred-Ahmadu et al., 2024; Aliyu et al., 2023). Everyday use of plastic containers for cooking and reheating therefore constitutes an additional, under-recognised dietary exposure route. Methodologically, the 10 % KOH digestion protocol proved effective for organic-matrix removal while preserving the integrity of PE and PP particles, corroborating previous validation studies (Thiele et al., 2019; Fred-Ahmadu et al., 2024). The dual FTIR–SEM strategy mitigates the limitations inherent to either technique alone: FTIR supplies polymer identity even when particle numbers are modest, while SEM supplies morphological confirmation and relative-abundance information.

Conclusion

This study investigated the occurrence and characteristics of suspected microplastics in four commonly consumed Nigerian foods (Tuwo, Moi-Moi, cake and Indomie) associated with plastic food-contact materials. FTIR spectra of food residues exhibited features consistent with those of the container reference material, while SEM revealed fibrous strands, irregular fragments and sheet-like particles with morphological characteristics consistent with suspected microplastics. Procedural blanks showed comparatively fewer polyolefin-associated features, supporting the importance of contamination controls during sample processing. However, the small sample size limits the generalisability of the findings, and the absence of quantitative particle counts and migration rates prevents definitive assessment of particle transfer from containers into food and associated dietary exposure. Limitations of the present work include the semi-quantitative nature of the abundance rankings (no particle-number normalisation per gram of food) and the focus on a single geographic area. Future studies should incorporate quantitative particle counting (e.g., by Raman or µ-FTIR mapping), polymer-mass estimation, and broader sampling across Nigeria to refine exposure estimates. Nevertheless, the qualitative and semi-quantitative evidence presented here is robust and demonstrates a clear container-to-food migration pathway under conditions typical of Nigerian household and restaurant practice. Further research should involve larger sample sizes, controlled experiments comparing food before and after contact with plastic containers, quantitative particle counting and standardised analytical protocols to establish the extent of potential microplastic transfer. Future studies should also assess potential chemical exposure and relevant health implications. Until exposure levels and associated risks are adequately quantified, public-health and regulatory recommendations should be guided by further scientific evidence.

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