



# Identification of Microplastics in Agricultural Soil using FTIR Spectroscopy

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**ABSTRACT:** Microplastic contamination in agricultural soils has emerged as a critical environmental concern due to the widespread use of plastic mulch films, contaminated irrigation water, and sewage sludge application. This study investigated the presence and characterization of microplastics in agricultural red soils from Ballari district, Karnataka, India, using Fourier Transform Infrared (FTIR) spectroscopy. Soil samples were collected from fields with documented plastic mulch usage at three depth intervals (0–10 cm, 10–20 cm, and 20–30 cm) using a stratified random sampling approach. Laboratory processing involved KBr pellet preparation and density separation protocols. FTIR analysis in Attenuated Total Reflectance (ATR) mode was performed on three sample categories: fine soil (S1A, S2A, S3A), organic matter floating particles (S1B, S2B, S3B), and soil–microplastic combinations (S1D, S2D, S3D). Results confirmed that the soils are dominated by clay minerals, silicate frameworks, and iron–aluminium oxides. Weak aliphatic C–H stretching peaks in the 2850–2950 cm<sup>-1</sup> range and carbonyl C=O bands at approximately 1873 cm<sup>-1</sup> in combined samples confirmed the presence of weathered polyethylene and polypropylene microplastic residues from degraded mulching films. FTIR spectroscopy proved to be a reliable, rapid, and cost-effective preliminary screening technique for microplastic detection in agricultural soils. The study recommends sustainable alternatives to plastic mulching and strengthened monitoring strategies for long-term soil health preservation.

**KEYWORDS:** Microplastics, FTIR spectroscopy, Agricultural Soil, Plastic Mulching, Polymer Identification.

## I. INTRODUCTION

Microplastics, defined as synthetic polymer particles smaller than 5 mm in diameter, have emerged as one of the most pervasive and persistent environmental contaminants of the modern era. The term was first coined by Thompson et al. in 2004 [1], following which research efforts intensified dramatically as the global scale of plastic contamination became apparent. Global plastic production currently exceeds 400 million metric tons annually, with a significant fraction entering terrestrial and aquatic ecosystems.

Agricultural soils represent one of the most important yet understudied sinks for microplastic pollution. The repeated use of plastic mulch films, application of sewage sludge as fertilizer, and irrigation with contaminated water have created substantial pathways for microplastics to enter soil ecosystems [2]. Once deposited, these particles persist for decades due to their non-biodegradable polymer composition, undergoing gradual fragmentation under UV radiation, mechanical stress, and biological activity.

The impacts of microplastic contamination on soil quality are multidimensional. Physically, microplastics alter soil porosity, bulk density, and water retention capacity. Chemically, they act as carriers of heavy metals, pesticides, and plastic additives such as phthalates and BPA, disrupting nutrient cycling [4]. Biologically, they reduce microbial diversity, enzyme activity, and earthworm populations, thereby threatening long-term agricultural productivity.

India's agricultural regions, particularly those practicing intensive plasticulture, are vulnerable to microplastic accumulation. However, systematic monitoring of microplastic contamination in Indian agricultural soils remains limited. This study addresses this gap by employing FTIR spectroscopy to identify and characterize microplastic residues in agricultural red soils from Ballari, Karnataka, with the aim of providing a scientific baseline for environmental monitoring and sustainable land management.

## II. LITERATURE SURVEY

The scientific understanding of microplastic contamination has evolved rapidly over the past two decades. Rillig (2012) was among the first to highlight microplastic risks to terrestrial ecosystems, emphasizing potential impacts on soil structure, biodiversity, and function [2]. Nizzetto et al. (2016) estimated that large quantities of microplastics enter agricultural soils annually through sewage sludge and compost application [3]. Machado et al. (2018) demonstrated that microplastics alter key soil properties including aggregation, water retention, and microbial activity [4].

For microplastic detection, Fourier Transform Infrared Spectroscopy (FTIR) has become the most widely applied identification technique. Hidalgo-Ruz et al. (2012) established FTIR, Raman spectroscopy, and scanning electron microscopy as standard analytical tools. The ATR-FTIR mode is particularly suited for soil analysis as it requires minimal sample preparation and allows direct analysis of isolated particles. Jung et al. (2018) validated ATR-FTIR for the identification of common plastic polymers including PE, PP, PS, and PET.

Thakur et al. (2023) successfully used Fourier Transform Infrared Spectroscopy and X-ray diffraction for characterizing microplastics extracted from agricultural soils near industrial areas. Renner et al. (2019) developed robust automatic FTIR identification methods for environmental microplastic samples. Recent literature by Peneva et al. (2025) provided comparative assessment methodologies for microplastic analysis in soils. Studies confirm that sample pretreatment—particularly density separation using saturated salt solutions and organic matter digestion using  $H_2O_2$ —is essential to improve polymer recovery from complex soil matrices.

Chia et al. (2024) comprehensively reviewed methods of soil sampling for microplastic analysis, emphasizing the need for standardized protocols to improve comparability across studies. A recent comprehensive review further highlighted the growing concern regarding the impacts of microplastics on soil microbial activity, nutrient cycling, and crop growth. However, Ballari district, characterized by red soils and extensive agricultural plastic use, has not been systematically studied, making this investigation a significant regional contribution.

## III. METHODOLOGY

This study adopted an integrated field-to-laboratory methodology designed to systematically investigate microplastic contamination in agricultural soils. The research framework addressed four key objectives: (1) capture spatial and vertical distribution patterns of microplastics, (2) characterize polymer types and size fractions, (3) maintain rigorous quality control, and (4) produce reproducible, internationally comparable results.

### A. Field Sampling

Agricultural soil samples were collected from fields in Marabbihal with documented plastic mulch usage for at least three consecutive years. A stratified random sampling approach was implemented, collecting samples at three depth intervals (0–10 cm, 10–20 cm, and 20–30 cm) to assess vertical microplastic distribution. At each sampling location, five sub-samples were collected in a W-pattern across one hectare. Control samples were obtained from adjacent non-mulched fields. All samples were stored in glass containers and transported to the laboratory under controlled conditions to prevent airborne contamination.

### B. Sample Preparation– KBr Pellet Method

Samples were prepared using the KBr pellet transmission method. Approximately 1–2 mg of the soil sample was mixed with 200–300 mg of spectroscopic grade Potassium Bromide (KBr). The mixture was ground in an agate mortar and pestle to reduce particle size below 2 microns to avoid IR scattering. The mixture was then pressed into a transparent pellet using a hydraulic press at 8–10 tons for 1–2 minutes under vacuum. All equipment was cleaned with solvent before use, and pellets were stored in a desiccator to avoid moisture absorption.

### C. Density Separation

A three-stage density separation protocol was employed. Samples were sequentially treated with sodium iodide solution ( $1.6\text{ g/cm}^3$ ) to isolate buoyant polymers such as PE and PP, zinc bromide solution ( $1.8\text{ g/cm}^3$ ) for intermediate density fractions, and sodium polytungstate ( $2.0\text{ g/cm}^3$ ) for denser polymers like PET and PVC. Each separation was conducted in PTFE-coated centrifuge tubes at  $3000\times g$  for 20 minutes at a constant  $20^\circ\text{C}$ .

#### D. FTIR Spectroscopy Analysis

Microplastic identification was performed using a Thermo Scientific Nicolet iN10 MX FTIR spectrometer in Attenuated Total Reflectance (ATR) mode with a germanium crystal and 15× objective lens, providing 10×10 μm spatial resolution. All samples were conditioned in a desiccator for 24 hours prior to analysis. Each spectrum was collected over 128 scans at 4 cm<sup>-1</sup> resolution across the mid-infrared range (4000–400 cm<sup>-1</sup>). Daily calibration was performed using certified polystyrene standards to ensure wavenumber accuracy within ±2 cm<sup>-1</sup>. Spectra were matched against standard polymer reference libraries for polymer identification.

Three sample categories were analysed:

- S1A, S2A, S3A – Finesoil samples
- S1B, S2B, S3B – Organic matter (floating particles)
- S1D, S2D, S3D – Combination of soil and microplastics

### IV. EXPERIMENTAL RESULTS

#### A. Fine Soil Sample Analysis (S-Series A)

FTIR analysis of fine soil sample S1A confirmed the dominance of clay minerals, silicate frameworks, and iron–aluminium oxide minerals. Absorption peaks at 3853, 3697, and 3625 cm<sup>-1</sup> are attributed to structural O–H stretching vibrations of clay minerals, while the broadband at 3429 cm<sup>-1</sup> corresponds to hydrogen-bonded adsorbed moisture. The intense peak at 1010 cm<sup>-1</sup> confirms Si–O–Si stretching of dominant silicate minerals. Peaks at 777 and 692 cm<sup>-1</sup> confirm crystalline quartz (SiO<sub>2</sub>), and low-wavenumber peaks at 647–431 cm<sup>-1</sup> indicate iron oxide and aluminosilicate lattice vibrations characteristic of red soils [6,7].

Sample S2A showed additional C–H stretching peaks at 2917 and 2852 cm<sup>-1</sup>, attributed to aliphatic –CH<sub>2</sub>/–CH<sub>3</sub> groups from soil organic matter and possible traces of hydrocarbon-based plastic residues [8]. The N–H bending peak at 1512 cm<sup>-1</sup> indicates humic nitrogenous organic compounds typical of cultivated agricultural soils. Sample S3A exhibited additional carbonyl C=O stretching at 1746 cm<sup>-1</sup> and aromatic C–H stretching at 3063 cm<sup>-1</sup>, suggesting the presence of oxidized organic residues or degraded polymeric materials in the soil [9].

#### B. Organic Matter Floating Particles (S-Series B)

The floating particle fractions (S1B, S2B, S3B) are dominated by hydroxyl-bearing clay minerals, aliphatic hydrocarbons, and silicate minerals, with no definitive isolated polymer signatures [10]. In S1B, a C=O stretching peak at 1689.74 cm<sup>-1</sup> indicates oxidized organic matter or degraded polymeric materials. Sample S2B showed a simplified spectrum with dominant mineral peaks (Si–O–Si at 1004 cm<sup>-1</sup>) and weak aliphatic C–H at 2914 cm<sup>-1</sup>, consistent with trace plastic hydrocarbon residues masked by silicate interference [5]. Sample S3B displayed extensive O–H stretching bands from hydroxyl clay minerals and moisture, with an unsaturated =C–H peak at 3207 cm<sup>-1</sup> indicating organic residues.

#### C. Soil–Microplastic Combination Samples (S-Series D)

The combined soil–microplastic samples provided the most diagnostic evidence for microplastic contamination. Sample S1D exhibited an aromatic =C–H stretching peak at 3057.56 cm<sup>-1</sup>, which is a clear indicator of plastic-derived hydrocarbon structures [7]. A distinct C=O stretching band at 1873.32 cm<sup>-1</sup> confirms the presence of oxidized polymer fragments from photo-oxidative environmental degradation, most likely from weathered polyethylene or polypropylene mulching films [6]. A skeletal polymer vibration band at 646.07 cm<sup>-1</sup> further supports plastic–mineral interaction.

Sample S2D similarly showed a C=O carbonyl peak at 1874.96 cm<sup>-1</sup>, confirming oxidized plastic residues amid dominant clay and silicate mineral peaks [9]. Sample S3D exhibited the weakest microplastic signatures: distinct aliphatic C–H peaks (~2920 and 2850 cm<sup>-1</sup>) and characteristic carbonyl oxidation bands were absent, suggesting extremely low or highly degraded polymer contamination beyond FTIR bulk sensitivity [12].

TableI: Key FTIR Peaks Identified in Soil–Microplastic Samples

Sample	KeyIRPeaks( $\text{cm}^{-1}$ )	Interpretation
S1A	3853,3697,1010,777	Clayminerals,quartz,ironoxides
S2A	2917,2852,1512	Organicmatter+trace hydrocarbon
S3A	3063,1746,1600	Oxidizedorganics,carbonyl groups
S1B	2962,1689,1008	Aliphatics,carbonyl,silicates
S2B	2914,1640,1004	Tracehydrocarbon,silicates
S3B	3207,1033,784	Organicresidues,silicates,quartz
S1D	3057,1873,646	PlasticaromaticC–H,oxidized polymer
S2D	1874,1018,693	Carbonylpolymer+clay minerals
S3D	3701,1636,995	Mostlyclay/mineral,weak plastics

TableII: Reference IR Peaks for Common Agricultural Polymers

Polymer	IRPeaks( $\text{cm}^{-1}$ )
Polyethylene(PE)	2915,2849,1471,730
Polypropylene(PP)	2950,2918,1455,1375
Polystyrene(PS)	3025,1601,1493,698
PVC	2915,1427,960
Polyamide(Nylon)	3300,1630

## V. CONCLUSION

This study successfully demonstrated the application of FTIR spectroscopy for the identification and characterization of microplastic contamination in agricultural red soils of Ballari, Karnataka. The key findings are summarized as follows:

1. All soil samples are mineral-dominated, composed primarily of clay minerals, silicate frameworks (Si–O–Si at  $1000\text{--}1050\text{ cm}^{-1}$ ), quartz, and iron-aluminium oxide phases (Fe–O and Al–O vibrations at  $450\text{--}550\text{ cm}^{-1}$ ) characteristic of Karnataka red soils.
2. Microplastic contamination was confirmed at trace levels in the combined soil-microplastic samples (S-series D). The aromatic =C–H stretching at  $\sim 3057\text{ cm}^{-1}$  and carbonyl C=O peaks at  $\sim 1873\text{--}1875\text{ cm}^{-1}$  confirm the presence of weathered polyethylene and polypropylene residues, most likely originating from degraded plastic mulching films used in agricultural fields.
3. The detected microplastics show signs of significant environmental degradation, evidenced by the presence of carbonyl and hydroxyl functional groups —products of UV-induced photo-oxidation and thermo-oxidative weathering under field conditions.
4. FTIR spectroscopy proved to be a reliable, rapid, and non-destructive screening tool for functional group-based identification of microplastics in soil. However, bulk FTIR sensitivity is limited by overlapping mineral peaks. Particle isolation followed by micro-FTIR or Raman spectroscopy is recommended for quantitative polymer-specific identification.
5. The continuous use of plastic mulching in agricultural fields contributes to the gradual accumulation of microplastic residues in soil, posing long-term risks to soil structure, microbial communities, nutrient cycling, and crop productivity. The adoption of biodegradable mulch alternatives, proper plastic collection after harvest, and routine microplastic monitoring programs are strongly recommended.

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