

# Occurrence and Distribution of Microplastics in Hydrologically Connected River–Reservoir Systems: Tepeji–Requena and Tula–Endhó, Mexico

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## Research Article

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# Abstract

Microplastics (MPs) were investigated in two hydrologically connected river–reservoir systems in central Mexico: the Tepeji River–Requena Reservoir and the Tula River–Endhó Reservoir. Water samples were processed by filtration and oxidative digestion, followed by optical microscopy for morphological and size analysis and micro-Raman spectroscopy for polymer identification. Plastic fragments were observed in all water bodies, mainly with sizes below 100  $\mu\text{m}$ , together with abundant microfibers with lengths of approximately 70–1300  $\mu\text{m}$ . Fiber concentrations were  $(4.0 \pm 0.5) \times 10^3$  and  $(6.0 \pm 0.8) \times 10^3$  fibers  $\text{L}^{-1}$  in the Tepeji and Tula rivers, respectively, increasing to  $(8.0 \pm 0.9) \times 10^3$  and  $(1.4 \pm 0.4) \times 10^4$  fibers  $\text{L}^{-1}$  in the Requena and Endhó reservoirs. Fragment concentrations were on the order of  $10^3$ – $10^4$  particles  $\text{L}^{-1}$  for particles  $\geq 20$   $\mu\text{m}$ , while the 1–20  $\mu\text{m}$  fraction of suspected microplastics was substantially more abundant. Raman analysis identified PET, PP, PE, and PA, with site-dependent distributions and spectral contributions associated with dyes in several fibers. The results suggest that the reservoirs may act as zones of enhanced MP accumulation within both connected systems. In situ physicochemical measurements and nonlinear optical laser-transmission experiments additionally revealed marked differences between sites, while similar optical responses provide complementary evidence of similarities in contaminant composition within the two connected systems, indicating that MP occurrence must be interpreted together with local hydrological and water-quality conditions.

## Introduction

Plastic debris has become a persistent contaminant in aquatic environments, and particles smaller than 5 mm are generally classified as microplastics (MPs). Although early studies mainly focused on marine systems, it is now clear that rivers, lakes, and reservoirs are important compartments for the transport, transformation, and temporary storage of MPs (Eerkes-Medrano et al., 2015; Range et al., 2023). Freshwater systems receive particles from municipal wastewater, industrial discharges, urban runoff, atmospheric deposition, agricultural activities, and the fragmentation of larger plastic products. Their abundance and spatial distribution are therefore controlled not only by the intensity of the sources but also by particle size, morphology, density, flow conditions, sedimentation, resuspension, and retention within reservoirs (Range et al., 2023; Wang et al., 2021). For this reason, the analysis of connected freshwater bodies is important for understanding how MPs can be transported and redistributed along a hydrological system rather than merely describing their occurrence at isolated sites.

A recurrent observation in freshwater studies is the high contribution of fibers and small fragments. Fibers are frequently associated with domestic and industrial textile sources, while fragments can be produced by the progressive mechanical and photochemical degradation of larger plastic objects. Polyethylene (PE), polypropylene (PP), polyethylene terephthalate (PET), and polyamide (PA) are among the polymers commonly reported in freshwater environments (Wang et al., 2021). However, direct comparisons among studies remain difficult because reported concentrations depend strongly on the sampled volume, mesh or filter size, digestion procedure, lower size limit, and spectroscopic confirmation method (Eerkes-Medrano et al., 2015; Prata, da Costa, Duarte, et al., 2019). This limitation is

especially important for particles below 100  $\mu\text{m}$ . As the analytical lower-size limit decreases, the number of detected particles can increase substantially, while visual identification also becomes less reliable. Consequently, studies that include micrometric particles can report concentrations that are not directly comparable to those from surveys based on meshes of several tens or hundreds of micrometers.

This methodological problem is also evident in Mexico. A recent systematic review by Caro-Martínez et al. (2025) evaluated 80 studies published between 2014 and 2024 across 20 Mexican states. The review showed that sediment, particularly beach sand, has been the most studied environmental matrix, followed by biota, water, and the atmosphere, indicating that the available information on aquatic environments remains comparatively limited. Fibers were among the most frequently reported morphologies, and PP and PE were among the polymers most commonly identified (Caro-Martínez et al., 2025). At the same time, the authors emphasized the substantial heterogeneity in sampling, particle-size limits, and identification procedures among Mexican studies. This situation highlights the importance of generating well-characterized freshwater datasets, particularly when small particles are included and the hydrological connectivity between sampling sites can be considered.

The Mezquital Valley in Hidalgo, central Mexico, represents a particularly relevant system for this type of study. Large volumes of wastewater generated in the Mexico City Metropolitan Area are transported to the valley and reused for agricultural irrigation. Treated and untreated wastewater streams interact with the Tula River and ultimately reach the Endhó Reservoir, and recent chemical studies have demonstrated the presence of a broad range of contaminants of emerging concern in this region (Caro-Martínez et al., 2025). The environmental relevance of the area has also been formally recognized by the Mexican Government. In September 2024, 36,637.93 ha within the area of influence of the Endhó Reservoir were declared an Ecological Restoration Zone (Zona de Restauración Ecológica, ZRE) because of accelerated environmental degradation and the need to restore ecological processes and ecosystem services (Secretaría de Medio Ambiente y Recursos Naturales, 2024). More recently, the National Water Commission (CONAGUA) implemented the Master Plan for the Sanitation and Recovery of the Endhó Reservoir, which includes sanitary control and environmental recovery actions (Comisión Nacional del Agua, 2026). These initiatives emphasize the need to characterize emerging contaminants, including microplastics, as part of the environmental assessment of these highly impacted aquatic systems.

Previous studies in Mexico provide useful points of comparison, although differences in environmental matrix and analytical size limits must be considered. Microplastics have been reported in freshwater sediments from the Atoyac River–Valsequillo Dam system in central Mexico (Shruti et al., 2019), while studies in Todos Santos Bay identified fibers and fragments in surface waters and wastewater-treatment-plant effluents and reported PE, PP, nylon, and other synthetic polymers (Ramírez-Álvarez et al., 2020). In stormwater runoff from Tijuana, median abundances of 66–191 particles  $\text{L}^{-1}$  were reported, with fibers and PE as the dominant components (Piñon-Colin et al., 2020). Microplastics have also been detected in drinking water from refill kiosks in the Mexico City metropolitan area at concentrations of 11–860 particles  $\text{L}^{-1}$ , with fibers being the most common morphology (Pérez-Guevara et al., 2022). These studies confirm the widespread occurrence of microplastics in Mexican aquatic environments, but

they also show that reported concentrations depend strongly on the sampled matrix, particle-size range, and analytical procedure.

Reservoirs are of special interest because reduced flow velocities and longer water residence times can modify the transport of suspended particles. Depending on particle density, shape, biofouling, and hydrodynamic conditions, a reservoir can act as a temporary sink, a transformation zone, or a secondary source during resuspension events (Range et al., 2023). Therefore, comparing a river with a hydraulically connected reservoir can provide information on changes in particle abundance and morphology along the same system. This approach is particularly useful for microfibers and small fragments, whose transport behavior can differ because of their geometry and effective settling characteristics. The Tepeji-Requena and Tula-Endhó systems provide an opportunity to make this comparison under different local water-quality and contamination conditions.

The objective of this study was to determine the occurrence, morphology, size distribution, and polymer composition of MPs and microfibers in the Tepeji River, Requena Reservoir, Tula River, and Endhó Reservoir. The main contribution of the work is the comparative analysis of two hydrologically connected river-reservoir systems, with particular attention to particles in the micrometric range that are commonly excluded by larger-mesh sampling methods. Optical microscopy was combined with micro-Raman spectroscopy for polymer identification (Chakraborty et al., 2023). In addition, dissolved oxygen (DO), oxidation-reduction potential (ORP), pH, temperature, and atmospheric pressure were measured in situ to provide physicochemical context for the samples. Finally, nonlinear optical transmission measurements at 532 nm were explored as a complementary characterization of the concentrated particulate suspensions. The results reveal a strong predominance of small fragments and greater microfiber abundance in both reservoirs than in their corresponding rivers, supporting the importance of considering hydrological connectivity when evaluating MP contamination in freshwater systems. Nonlinear optical characterizations also offer additional evidence of the connection between these systems and provide an initial approach toward an optical method for the characterization of MPs in real environmental samples.

## **Materials and Methods**

### **Study area and sampling**

The study was carried out in southern Hidalgo, Mexico, across two hydrologically connected systems: (i) the Tepeji River and Requena Reservoir, and (ii) the Tula River and Endhó Reservoir. The four sites are located in a region with intense urban, industrial, and agricultural activity. The Tula system additionally receives a major contribution from wastewater conveyed from the Mexico City Metropolitan Area to the Mezquital Valley (Garduño-Jiménez et al., 2023). The geographical location of the four sampling sites is shown in Fig. 1.

Surface-water samples were collected from accessible sampling locations in the Tepeji River, Requena Reservoir, Tula River, and Endhó Reservoir. At each sampling site, three independent field replicates were collected. A volume of 500 mL was collected for each replicate at approximately 30 cm below the water surface. The three replicates were collected separately and subsequently processed independently following the same sample-preparation procedure. The geographical coordinates of each sampling location are provided in Fig. 1. The reported microplastic and microfiber concentrations correspond to the mean values obtained from the three independent field replicates. Error bars were estimated from the standard deviation of the three independent field replicates.

Particular care was taken to minimize exposure of the samples to plastic materials during processing whenever possible. To minimize potential contamination during sample handling and processing, several precautionary measures were implemented. Glassware was used whenever possible, nitrile gloves were worn throughout sample processing, and samples were exposed to the laboratory environment only when necessary. All glassware and materials in contact with the samples were thoroughly cleaned before use.

## Sample preparation and isolation of microplastics

Water samples were initially pre-filtered through a 2 mm plastic mesh to remove large organic matter and coarse debris, thereby facilitating the subsequent sample-processing steps. The material recovered was subjected to oxidative digestion using Fenton's reagent to remove residual organic matter. A 50 mM  $\text{Fe}^{2+}$  solution was prepared by dissolving 1.39 g of iron(II) sulfate heptahydrate [ $\text{Fe}(\text{SO}_4) \cdot 7\text{H}_2\text{O}$ ] in distilled water and adjusting the final volume to 100 mL. The solution was stored away from light until use.

For oxidative digestion, 25 mL of 30% (w/w) hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) and 1 mL of the  $\text{Fe}^{2+}$  solution were slowly added to 20 mL of each previously processed sample. The mixtures were maintained under continuous stirring at 40°C for 1 h. After digestion, the samples were allowed to cool to room temperature.

Following oxidative digestion, each sample was filtered to recover the remaining particulate material for microplastic analysis. Water samples were processed by vacuum filtration using a Rocker 300 vacuum pump. Vacuum filtration was performed using nylon meshes with pore sizes of 149 and 60  $\mu\text{m}$ . The microfibers recovered from the samples were retained on the nylon meshes, which were subsequently used directly for microscopic observation and microfiber counting. Importantly, the fraction passing through the 60  $\mu\text{m}$  nylon mesh was not discarded. The filtrate containing particles smaller than the mesh pore size was collected and subsequently concentrated by centrifugation at 4000 rpm for 20 min. A 1 mL concentrated suspension was retained for microscopic analysis. A 50  $\mu\text{L}$  aliquot was deposited onto a clean glass microscope slide and dried under covered conditions. Therefore, particles in the 1–10 and 10–20  $\mu\text{m}$  size ranges were quantified from the fraction passing through the 60  $\mu\text{m}$  mesh rather than from particles retained on the mesh. Fenton-assisted oxidation has been previously shown to provide

rapid removal of organic-rich matrices while maintaining the chemical identification of common polymers (Tagg et al., 2017; Prata, da Costa, Girão, et al., 2019).

To minimize potential contamination during sample preparation and analysis, glass materials were cleaned with distilled water and subjected to ultrasonic cleaning before use. Clean glass slides were stored in covered Petri dishes until sample deposition, and samples were kept covered whenever possible during laboratory processing. Procedural blanks consisting of distilled water were subjected to the same sample-preparation procedure as the environmental samples, including Fenton digestion, vacuum filtration, centrifugation, deposition onto glass slides, and microscopic examination. Microfiber- and microplastic-like particles detected in the procedural blanks represented less than 10% of those measured in the environmental samples, indicating a relatively minor contribution from procedural contamination. For microfibers retained on the nylon meshes, the micro-Raman laser was focused directly on the selected fibers to minimize the spectral contribution from the filtration substrate. The nylon filtration mesh was characterized separately so that its spectral contribution could be distinguished from that of the environmental fibers. Particles were classified as suspected MPs prior to Raman confirmation based on their morphology, surface appearance, and absence of obvious biological structures. Since visual classification becomes less reliable for very small particles, the 1–20 µm population was discussed separately from the more conservative ≥ 20 µm fraction.

## Optical microscopy, morphology, and size distribution

Dried aliquots and retained material were inspected by optical microscopy at different magnifications using a Keyence VHX-7000 High Resolution Digital Microscope. Particles were grouped into four morphological categories: fragments, fibers, film-like particles, and rounded/pellet-like particles. The characteristic dimension of compact particles was used for the size distribution, with bins of 1–10, 10–20, 20–50, 50–100, and 100–200 µm. Fibers were evaluated separately because their elongated morphology is not adequately represented by the same bins; observed fiber lengths were approximately 70–1200 µm. Concentrations were calculated by accounting for the number of particles counted, the fraction of the deposited area examined, the aliquot volume, the final concentrated sample volume, and the original volume of water processed, and were reported as particles L<sup>-1</sup> or fibers L<sup>-1</sup>. Microplastics concentrations were calculated according to

$$C = \frac{N}{f_A} \frac{V_c}{V_a} \frac{1}{V_s}$$

1

where  $C$  is the particle or microfiber concentration (particles L<sup>-1</sup> or fibers L<sup>-1</sup>),  $N$  is the number of particles or fibers counted within the analyzed area,  $f_A$  is the fraction of the total deposited area examined,  $V_c$  is the final concentrated suspension volume (1 mL),  $V_a$  is the volume of the aliquot

deposited onto the glass slide, and  $V_s$  is the original volume of water represented by the processed sample, expressed in liters.

## Micro-Raman spectroscopy

Micro-Raman measurements were performed using a Horiba Scientific XploRA PLUS Raman spectrometer with 532 nm laser excitation. Spectra were acquired using  $5\times$  and  $10\times$  VIS objectives, depending on particle size and morphology, with the laser power attenuated to 1% of the available output. An acquisition time of 1 s with 50 accumulations was used. Measurements were performed using a 600 grooves  $\text{mm}^{-1}$  grating, a 200  $\mu\text{m}$  slit, and a 500  $\mu\text{m}$  confocal hole, over a spectral range of 100–3499  $\text{cm}^{-1}$ . Spectra were acquired over the fingerprint and C–H stretching regions and compared with characteristic Raman bands reported for common polymers. Assignments were made primarily to PET, PP, PE, and PA. Raman spectroscopy is well established for micrometric MP identification, but environmental weathering, pigments, fluorescence, and additives can modify band intensities and background profiles (Chakraborty et al., 2023; Phan et al., 2022). For this reason, polymer assignment was based on groups of diagnostic bands reported in the literature rather than on a single peak. Peaks that did not correspond to the polymer backbone and were prominent in colored fibers were treated as possible dye-related contributions. At least 30 individual particles and fibers from each sampling site were analyzed by micro-Raman spectroscopy. Particles were selected to represent the different morphologies, sizes, and colors observed in each sample. The relative polymer composition was estimated from the proportion of particles identified as each polymer type relative to the total number of particles successfully characterized by Raman spectroscopy.

## In-situ physicochemical measurements

Physicochemical parameters were recorded in situ at the time of water sampling using a Hanna Instruments HI98196 multiparameter probe. Dissolved oxygen and ORP were monitored for tens of seconds until a stable response was obtained. Temperature, pH, and atmospheric pressure were recorded simultaneously. These measurements were not used as direct indicators of MP abundance; rather, they were included to describe the markedly different physicochemical environments in which the particles were transported and retained.

## Nonlinear optical transmission

Concentrated suspensions containing the isolated particulate fraction were placed in 1 cm optical cells and irradiated at 532 nm with laser pulses of approximately 29 ps duration generated by the second harmonic of an EKSPLA laser, with pulse energies ranging from 38 to 500  $\mu\text{J}$ . The incident beam diameter at the sample was approximately 0.1 mm. The transmitted signal was measured as a function of the incident pulse energy using a typical input-output configuration (Bornacelli et al., 2026). The

nonlinear absorption coefficient for each sample was obtained by fitting the data using the following equation (Bornacelli et al., 2026):

$$I(L) = \frac{I_0 e^{-\alpha_0 L}}{1 + \beta I_0 L_{eff}}$$

2

where  $I(L)$  and  $I_0$  are the transmitted and incident irradiances, respectively. The linear and nonlinear absorption coefficient are  $\alpha_0$  and  $\beta$ , respectively. Here,  $L_{eff} = (1 - e^{-\alpha_0 L})/\alpha_0$ , where  $L$  is the actual sample thickness. The experimental values of  $L$  and  $\alpha_0$  were obtained from the 1 cm optical path length of the cuvette and the linear optical absorption spectrum of each sample, respectively. At low and intermediate energies, the transmission response was analyzed through its approximately linear trend. For the Tula and Endhó samples, a departure from this trend was observed above a characteristic incident-energy interval. Because the measurements were performed on heterogeneous environmental suspensions, this change is conservatively referred to here as a laser-induced thermal/photothermal threshold response of the samples.

## Results

Microplastic-like particles and fibers were observed in all four water bodies. Figures 2–5 summarize representative optical images together with the morphology and size distributions. The dominant feature common to the four sites is the large number of small suspected microplastic fragments, particularly below 20  $\mu\text{m}$ . In contrast, the fiber fraction is numerically smaller than the fragment fraction but remains environmentally important because fibers were consistently detected at all sites and reached millimeter-submillimeter lengths.

## Morphology and size distribution

At the Tepeji River, fragments were the dominant morphology, with a concentration close to  $(4.5 \pm 0.8) \times 10^4$  fragments  $L^{-1}$  for the morphologically classified fraction, while fibers had a concentration of approximately  $(4.0 \pm 0.5) \times 10^4$  fibers  $L^{-1}$ . Approximately, 60% of the observed microfibers were transparent, however, Figs. 2c)-f) show microfibers with red, dark, blue and orange coloration, which may be associated with the presence of dyes or pigments. Film-like and rounded/pellet-like particles represented only minor fractions. The size histogram shows that the abundance increased strongly toward the smallest size classes. The 1 – 10  $\mu\text{m}$  class reached approximately  $(4.3 \pm 0.9) \times 10^4$  particles  $L^{-1}$  and the 10 – 20  $\mu\text{m}$  class was about  $(7.6 \pm 1.8) \times 10^4$  particles  $L^{-1}$ , whereas concentrations decreased to the  $10^4 - 10^3$   $L^{-1}$  range above 20  $\mu\text{m}$ . On the other hand, the microfiber concentration was of  $(4.0 \pm 0.5) \times 10^3$  fibers  $L^{-1}$ . The lengths of the microfibers ranged from 70 to 830  $\mu\text{m}$ .

The Requena Reservoir showed the same general pattern but lower fragment concentrations than the Tepeji River. Fragments were approximately  $(1.2 \pm 0.5) \times 10^4 L^{-1}$ , film-like particles about  $(2.0 \pm 0.3) \times 10^4 L^{-1}$ , and microfibers had a concentration of  $(8.0 \pm 0.9) \times 10^3 \text{ fiber } L^{-1}$ . The lengths of the microfibers ranged from 140 to 490  $\mu m$ , with a lower mean length than that observed in the Tepeji River. Approximately, 80% of the observed microfibers were transparent, while the remaining fibers exhibited various colors, including dark, blue, and orange, as shown in Figs. 3c)-f). These colorations may be associated with the presence of dyes or pigments. Rounded/pellet-like particles were scarce. The smallest class again dominated, with approximately  $(9.0 \pm 1.7) \times 10^4 \text{ particles } L^{-1}$  at  $1 - 10 \mu m$  and around  $(2.0 \pm 0.4) \times 10^4 L^{-1}$  at  $10 - 20 \mu m$ . Although the number of small fragments was lower than in the Tepeji River, the microfiber concentration increased in the reservoir, suggesting that transport and retention may differ between compact fragments and elongated fibers.

In the Tula River, fragments were also the dominant morphology, at approximately  $(3.4 \pm 0.4) \times 10^4$  fragments  $L^{-1}$ , followed by fibers at  $(6.0 \pm 0.8) \times 10^4 \text{ fibers } L^{-1}$ . The lengths of the microfibers ranged from 198 to 1300  $\mu m$ . Film-like and rounded/pellet-like particles each contributed less than  $(2.0 \pm 0.3) \times 10^4 L^{-1}$ . The  $1 - 10$  and  $10 - 20 \mu m$  fractions were approximately  $(3.3 \pm 0.6) \times 10^4$  and  $(6.0 \pm 1.4) \times 10^4 \text{ particles } L^{-1}$ , respectively. The concentration above 50  $\mu m$  decreased sharply, and particles larger than 100  $\mu m$  were rare. This distribution is consistent with a highly fragmented particulate population rather than a population dominated by large primary MPs. Approximately, 40% of the observed microfibers were transparent, while the remaining fibers exhibited various colors, including green, dark, and orange, as shown in Figs. 4c)-f). These colorations may be associated with the presence of dyes or pigments.

The Endhó Reservoir presented the highest microfiber concentration among the four sites, approximately  $(1.4 \pm 0.4) \times 10^5 \text{ fibers } L^{-1}$ . Approximately, 80% of the observed microfibers were transparent, while the remaining fibers exhibited various colors, including red, and orange, as shown in Figs. 5c)-f). These colorations may also be associated with the presence of dyes or pigments. The lengths of the microfibers ranged from 148 to 560  $\mu m$ . Fragments were also abundant, with a concentration close to  $(5.0 \pm 1.2) \times 10^4 L^{-1}$ , while rounded/pellet-like particles were approximately  $(8.0 \pm 1.7) \times 10^4 L^{-1}$  and film-like particles were a minor fraction. The  $1 - 10 \mu m$  population exceeded  $(5.0 \pm 1.2) \times 10^4 \text{ particles } L^{-1}$  and the  $10 - 20 \mu m$  population was close to  $(8 \pm 2.2) \times 10^4 L^{-1}$ . For the more conservative  $\geq 20 \mu m$  fraction, the concentration was of the order of  $(6.0 \pm 1.3) \times 10^4 L^{-1}$ . As at the other sites, particles larger than 100  $\mu m$  were scarce, and no substantial population above 200  $\mu m$  was observed.

When the two connected systems are considered together, higher mean microfiber concentrations were observed in both reservoirs than in their corresponding rivers, increasing from  $4 \times 10^3$  to  $8 \times 10^3 \text{ fibers } L^{-1}$  in the Tepeji–Requena system and from  $6 \times 10^3$  to  $1.4 \times 10^4 \text{ fibers } L^{-1}$  in the Tula–Endhó system. The pattern for fragments was less uniform, emphasizing that reservoir retention cannot be described by a single concentration factor across all particle morphologies.

# Polymer identification by Raman spectroscopy

Representative Raman spectra are shown in Fig. 6. The spectra confirm that the visually selected particles include common synthetic polymers. PET, PP, PE, and PA were identified across the complete set of samples, although their relative occurrence varied among sites and among the selected particles. PET-rich spectra showed the expected aromatic and ester-related fingerprint features together with C–H stretching bands, while PP and PE were identified by their characteristic C–C, CH<sub>2</sub>/CH<sub>3</sub> deformation, and stretching modes. Several colored fibers presented additional intense bands that could not be assigned solely to the polymer backbone and were therefore attributed to dyes or pigments associated with the fibers. This was the case in Fig. 6a) and d) where additional Raman bands not attributable to the identified polymers were consistent with synthetic organic pigments. In particular, bands observed in the Tepeji microfiber showed similarities to those of copper phthalocyanine (C.I. Pigment Blue 15) (Perraki et al., 2024), whereas the Endhó spectrum exhibited bands tentatively consistent with the dioxazine pigment C.I. Pigment Violet 23 (Nava et al., 2021). Partial correspondence was also observed with Raman bands reported for dyes such as Crystal Violet and Methylene Blue (Kumar & Boruah, 2014; Angeloni et al., 1979). However, because several of these compounds exhibit overlapping Raman bands and the spectra of environmental particles may contain simultaneous contributions from polymers, pigments, additives, surface contaminants, and substrate, the specific dyes cannot be unequivocally identified from the present measurements. Therefore, these bands are conservatively assigned to pigment/dye-related contributions rather than to a specific coloring compound.

The inset distributions in Fig. 6 indicate a site-dependent polymer composition. Some analyzed subsets were dominated by PET, whereas others showed a higher contribution of PE or a mixed PP/PE composition. This heterogeneity is expected for environmental MPs because the sampled particles originate from multiple consumer and industrial sources and can be modified by weathering. The Raman assignments should therefore be interpreted as the composition of the spectroscopically analyzed subset, rather than an absolute polymer census of every particle counted by microscopy.

## Physicochemical conditions

Figure 7 shows the in-situ ORP and DO measurements. The Tepeji River showed an alkaline pH of 8.8 at 19.8 °C and a stable positive ORP near +145 mV, with DO close to 60% saturation. Requena Reservoir was more alkaline (pH 9.7; 22.3 °C), with ORP ranging from +95 to +100 mV and DO near 60%. The Tula River showed a pH of 7.7 at 13.7 °C, a positive ORP of approximately +65 to +75 mV, and considerably lower DO, near 20–25%. The strongest contrast was observed at Endhó Reservoir (pH 7.4; 17.4 °C), where ORP was strongly negative, decreasing from approximately –325 to –365 mV, and DO approached very low values. These data indicate reducing, oxygen-poor conditions at Endhó at the time of sampling.

**Figure 7** In situ oxidation–reduction potential (ORP) and dissolved oxygen (DO) measured during sampling at the (a) Tepeji River, (b) Requena Reservoir, (c) Tula River, and (d) Endhó Reservoir.

Temperature, atmospheric pressure, and pH are indicated in each panel

The physicochemical measurements do not establish a causal relationship with MP concentration, but they demonstrate that particles are being transported through water bodies with very different redox conditions. In particular, the combination of high microfiber abundance and reducing conditions at Endhó suggests that particle accumulation occurs in a reservoir that also receives a substantial organic and wastewater-derived load.

## Laser-transmission response

The concentrated microplastics particulate suspensions also showed different transmission responses under 532 nm picosecond excitation (Fig. 8). For the Tepeji and Requena samples, transmission decreased approximately linearly over most of the incident-energy range explored as shown in Fig. 8a). Requena exhibited a higher low-energy transmission than Tepeji, but both curves converged toward lower transmission as the pulse energy increased. This behavior is consistent with intensity-dependent attenuation in a heterogeneous suspension, in which absorption and scattering can both contribute.

A different response was observed for the Tula and Endhó samples. At low incident energies, transmission initially increased, reaching values around 0.83–0.86. Above approximately 220–280  $\mu\text{J}$ , both samples showed an abrupt departure from the low-energy trend and a subsequent decrease in transmission. The shaded region in Fig. 8b) is therefore designated as a thermal threshold. Considering the 29 ps pulse duration and the small illuminated area, localized heating, bubble formation or other photothermal modifications could produce a sudden change in transmitted intensity. The estimated nonlinear optical absorption coefficient,  $\beta$ , was of  $2.9 \times 10^{-3}$  and  $2.8 \times 10^{-3}$  cm/GW for the Tepeji River and Requena Reservoir, respectively. On the other hand,  $\beta$  has a value of  $-3 \times 10^{-3}$  and  $-8 \times 10^{-3}$  cm/GW for the Tula River and Endho Reservoir, respectively.

## Discussion

The results demonstrate that MPs are widely distributed in both studied river–reservoir systems and, more importantly, that the measured abundance is strongly controlled by the lower size limit of the analysis. At every site, the  $1 - 10 \mu\text{m}$  and  $10 - 20 \mu\text{m}$  size classes were much more abundant than particles above  $20 \mu\text{m}$ . This observation agrees with the general expectation that progressive fragmentation shifts environmental plastic debris toward smaller dimensions, but it also illustrates a methodological problem: studies based on coarse nets or filters cannot be directly compared with microscopy-based measurements extending down to a few micrometers (Eerkes-Medrano et al., 2015; Prata, da Costa, Duarte, et al., 2019). In addition, reliable micro-Raman identification was particularly challenging for particles within the  $1-20 \mu\text{m}$  size range. For this reason, the present study classifies the  $< 20 \mu\text{m}$  population as suspected MPs and gives greater interpretive weight to the  $\geq 20 \mu\text{m}$  fraction supported by Raman measurements.

The dominance of fragments is consistent with secondary MPs generated by the degradation of larger plastic materials. At the same time, the consistent occurrence of microfibers points to an important contribution from textile and wastewater-related sources. Reviews of freshwater environments have identified fibers smaller than 1 mm as one of the most common MP morphologies and have linked high MP loads in impacted areas to sewage discharges (Wang et al., 2021). Similar patterns have been reported in Mexico: fibers represented an important fraction of MPs in surface waters and WWTP effluents from Todos Santos Bay (Ramírez-Álvarez et al., 2020), were the dominant morphology in stormwater runoff from Tijuana (Piñon-Colin et al., 2020), and were also the most common particles in drinking water from refill kiosks in the Mexico City metropolitan area (Pérez-Guevara et al., 2022). The concentrations reported for Tijuana (66–191 particles L<sup>-1</sup>) and drinking-water kiosks (11–860 particles L<sup>-1</sup>) are substantially lower than the concentrations measured here; however, a direct numerical comparison is not appropriate because the matrices, sampling strategies, and lower particle-size limits differ among studies. This is particularly relevant to the present work, in which the abundance rises strongly toward the micrometric size classes. The Mezquital Valley receives large wastewater flows from the Mexico City Metropolitan Area, and both treated and untreated streams interact with the river and irrigation network (Caro-Martínez et al., 2025). In the Tepeji River, textile-related wastewater discharges provide an additional plausible local source of synthetic fibers. These source associations are consistent with the observations, although source apportionment would require a larger polymer-resolved dataset and direct sampling of individual discharges.

The comparison between each river and its reservoir provides one of the most important results of this work. Microfiber concentration increased from  $4 \times 10^3$  fibers L<sup>-1</sup> in the Tepeji River to  $8 \times 10^3$  fibers L<sup>-1</sup> in Requena Reservoir, and from  $6 \times 10^3$  fibers L<sup>-1</sup> in the Tula River to  $1.4 \times 10^4$  fibers L<sup>-1</sup> in Endhó Reservoir. Reservoirs can modify MP transport through lower flow velocities, longer residence times, settling, biofouling, shoreline trapping, and episodic resuspension. Current hydro-geomorphic analyses emphasize that rivers and reservoirs cannot be treated simply as passive conduits and that particle size, shape, and density influence their transport pathways (Range et al., 2023). The higher fiber concentrations in both reservoirs are compatible with enhanced retention or accumulation, although the available data do not allow a residence-time or mass-balance calculation. The occurrence of MPs in the Atoyac River–Valsequillo Dam system (Shruti et al., 2019) provides a relevant central Mexico precedent for microplastic contamination in a connected river–reservoir setting, although that study analyzed sediments rather than the water column. Comparable patterns have been reported in river–reservoir systems from other countries. In the Aras River and Reservoir in Iran, surface-water concentrations reached 43 items m<sup>-3</sup> and the reservoir was proposed as a potential sink for MPs (Vayghan et al., 2022). In the Jiayan Reservoir in China, considerably higher concentrations of  $1.10 \times 10^4$ – $6.17 \times 10^4$  items m<sup>-3</sup> ( $11$ – $61.7$  items L<sup>-1</sup>) were measured, with small MPs ( $< 300 \mu m$ ) representing about 80% of the particles and PE fragments dominating the assemblage (Niu et al., 2022). A source-water river–reservoir system in central China showed  $3$ – $7.02$  items L<sup>-1</sup> in river water and  $2.07$ – $14.28$  items L<sup>-1</sup> in reservoir water, with fibers  $< 0.5 mm$  and PP and PE as the predominant morphologies and polymers, respectively (Shen et al., 2025). Similarly, Hirakud Reservoir in

India contained 82–89 particles  $L^{-1}$  in surface water, with fibers accounting for 46.21% and particles of  $53 - 300 \mu m$  forming the dominant size fraction (Patra & Baitharu, 2024). These international values are lower than the concentrations obtained in the present study, particularly for Endhó and Requena. However, this difference should not be interpreted directly as a proportional difference in pollution levels, because sampling volumes, particle-size cutoffs, sample preparation, and identification criteria differ substantially among studies. Nevertheless, the international comparisons support two features observed here: reservoirs can act as zones of MP retention, and the measured abundance increases markedly when smaller particle-size fractions are included.

Although many freshwater studies have reported substantially lower MP concentrations, abundances comparable to those measured in the present study have also been observed in highly impacted river systems, particularly when smaller particle-size fractions are considered. In the Yamuna River, India, Pawak et al. (2024) reported an average concentration of  $14,717 \pm 4,444$  MPs  $L^{-1}$ , with values reaching approximately 27,000 MPs  $L^{-1}$  at individual sites; notably, most particles were within the 2–80  $\mu m$  size range. Similarly, concentrations of 4,500–5,130 MPs  $L^{-1}$  have been reported in water from the lower Yangtze River, China (Han et al., 2024). These concentrations are of the same order of magnitude as those measured in the present study and demonstrate that MP abundances in the range of  $10^3$ – $10^4$  particles  $L^{-1}$  have been reported in highly impacted freshwater environments. Nevertheless, direct quantitative comparisons should be made cautiously because reported abundances are strongly influenced by sampling volume, particle-size detection limits, sample preparation, and identification methodology. This distinction is particularly relevant for some of the river–reservoir studies discussed above, such as the Atoyac River–Valsequillo Dam system (Shruti et al., 2019), where MPs were quantified in sediments rather than in the water column.

The polymer assignments also provide useful information about potential sources. PE and PP are high-volume polyolefins used in packaging and many disposable products, whereas PET and PA are extensively used in textile fibers and consumer materials. The coexistence of these polymers is therefore expected in waters receiving mixed urban and industrial inputs. Raman analysis of environmental MPs is nevertheless complicated by weathering and pigments. Controlled studies have shown that environmental aging modifies the Raman response of PE and PP, while PET and PA can also exhibit spectral changes after weathering (Phan et al., 2022; Phan et al., 2026). In the present spectra, dye-related peaks were especially evident in several fibers. These peaks should not be treated as evidence against a plastic origin; rather, they reflect the composite nature of colored textile fibers, in which the polymer substrate and dye molecules contribute simultaneously to the measured spectrum.

The in-situ water-quality measurements show that the two systems were not chemically equivalent at the time of sampling. Tepeji and Requena were alkaline and moderately oxygenated, whereas the Tula River had lower DO and Endhó displayed strongly reducing conditions and very low DO. The Endhó result is consistent with a water body receiving a substantial organic and wastewater load. Previous work in the Mezquital Valley has documented the transport of wastewater-derived contaminants and the complex mixture of treated and untreated flows entering the regional hydrological network (Garduño-

Jiménez et al., 2023). The coincidence of high microfiber abundance with reducing conditions at Endhó does not prove that the redox state controls MP accumulation, but it reinforces the interpretation of the reservoir as an integrated receiving compartment for both particulate and dissolved and organic contamination.

The laser-transmission experiment was included as an exploratory and complementary characterization. The nearly monotonic decrease in transmission observed for Tepeji and Requena differs from the threshold-like response exhibited by Tula and Endhó. Because these samples are heterogeneous suspensions containing different polymer types and particle sizes, a single microscopic nonlinear-absorption mechanism cannot be assigned from transmission measurements alone. However, if a particular polymer dominates the particle population in a given sample, its optical properties may contribute significantly to the observed response under intense laser irradiation.

Interestingly, micro-Raman analysis indicated a higher relative abundance of PE in the Tula and Endhó samples than in Tepeji and Requena. This compositional difference may contribute to the distinct optical response observed in these samples. Among the main polymers identified in the present study, PE exhibits a lower melting temperature than PP and PET; representative melting temperatures of approximately 141, 179, and 264°C have been reported for PE, PP, and PET, respectively (Prosenc et al., 2021). Therefore, the higher relative abundance of PE in Tula and Endhó could increase the susceptibility of the suspended microplastic particles to laser-induced heating, softening, or melting under intense, pulsed irradiation. Nevertheless, this interpretation should be regarded as a plausible contributing factor rather than direct evidence of a polymer-specific thermal transition. In the Tula and Endhó samples, the abrupt change observed at pulse energies of approximately  $220 - 280 \mu J$  is therefore more cautiously interpreted as the onset of laser-induced thermal or photothermal alteration. The use of the term *ablation* would require independent evidence of material removal, crater formation, plume generation, or permanent post-irradiation damage. This distinction is important because low-melting polyolefins such as PE and PP can undergo softening or melting at substantially lower temperatures than those required for true ablative decomposition.

## Conclusions

Microplastics and microfibers were detected in the Tepeji River, Requena Reservoir, Tula River, and Endhó Reservoir. The particulate population was dominated by small fragments, with a strong increase in abundance below  $20 \mu m$ , while fibers with lengths of approximately  $70-1300 \mu m$  were present at all sites. Microfiber concentrations increased from the river to the reservoir in both connected systems, reaching  $8 \times 10^3$  fibers  $L^{-1}$  in Requena and  $1.4 \times 10^4$  fibers  $L^{-1}$  in Endhó. Raman spectroscopy confirmed PET, PP, PE, and PA and revealed dye-related contributions in several fibers. Endhó combined the highest microfiber concentration with strongly reducing and oxygen-poor conditions, illustrating the multiple environmental pressures acting on this reservoir. The 532 nm nonlinear optical transmission measurements further differentiated the samples and provided additional evidence of similarities between samples within the hydrologically connected system. Pulsed laser irradiation induced a thermal

threshold in the Tula and Endhó suspensions, and this characteristic signal can be associated with the higher relative abundance of PE observed in these samples. These optical measurements represent an initial approach toward the development of an optical method for the characterization of MPs in real environmental samples. Our findings suggest that pulsed optical transmission may provide complementary information on heterogeneous microplastic-containing suspensions. Further studies using controlled polymer suspensions, calibration procedures, and validation against established spectroscopic techniques are required to evaluate its potential as an analytical approach. Overall, the results show that river–reservoir connectivity and the analytical lower-size limit are important factors to consider when evaluating MP contamination in the Tula–Tepeji region. Future work should extend temporal sampling and increase the fraction of particles confirmed spectroscopically.

## **Declarations**

### **Ethical responsibility of authors**

All authors have read and approved the final manuscript. The authors declare that this work is original, has not been published previously, is not under consideration for publication elsewhere, and complies with the ethical standards of Environmental Monitoring and Assessment. All authors have read, understood, and have complied as applicable with the statement on “Ethical responsibilities of Authors” as found in the Instructions for Authors.

### **Competing interests**

The authors declare no competing interests.

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## **Author Contribution**

Sampling, data curation, writing—original draft preparation J.A.B.; Conceptualization, methodology, writing review and editing L.M.A and H.M.A.; Formal analysis, writing-editing, methodology A.O. and J.A.R.E.; Conceptualization, investigation, methodology, visualization, formal analysis, supervision, writing-editing J.B., All authors have read and agreed to the published version of the manuscript.

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## Data Availability

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

## References

1. Angeloni, L., Smulevich, G., & Marzocchi, M. P. (1979). Resonance Raman spectrum of crystal violet. *Journal of Raman Spectroscopy*, 8, 305–310. <https://doi.org/10.1002/jrs.1250080603>
2. Bornacelli, J., Robledo-Hernández, C., Montiel-Rodríguez, A. Y., Martínez-Ayala, L., Martines-Arano, H., Torres-Torres, C., Reyes-Esqueda, J. A., & Oliver, A. (2026). Ultraviolet third-order nonlinear optical response from light-emitting copper nanoclusters embedded in silica. *Journal of Physics D: Applied Physics*, 59, 035303. <https://doi.org/10.1088/1361-6463/ae329f>
3. Caro-Martínez, D. M., Niño-Torres, C. A., Charruau, P., Rendón-von Osten, J., Castelblanco-Martínez, D. N., Rios Mendoza, L. M., Frausto-Martínez, O., & Blanco-Parra, M. D. P. (2025). The state of microplastic pollution in México: A review and evolving perspectives. *Science of the Total Environment*, 988, 179772. <https://doi.org/10.1016/j.scitotenv.2025.179772>
4. Chakraborty, I., Banik, S., Biswas, R., Yamamoto, T., Noothalapati, H., & Mazumder, N. (2023). Raman spectroscopy for microplastic detection in water sources: A systematic review. *International Journal of Environmental Science and Technology*, 20, 10435–10448. <https://doi.org/10.1007/s13762-022-04505-0>
5. Comisión Nacional del Agua. (2026, May 12). *Plan Maestro de Saneamiento y Recuperación de la Presa Endhó [Master Plan for the Sanitation and Recovery of the Endhó Dam]*. Gobierno de México. <https://www.gob.mx/conagua/prensa/plan-maestro-de-saneamiento-y-recuperacion-de-la-presa-endho>
6. Eerkes-Medrano, D., Thompson, R. C., & Aldridge, D. C. (2015). Microplastics in freshwater systems: A review of the emerging threats, identification of knowledge gaps and prioritisation of research needs. *Water Research*, 75, 63–82. <https://doi.org/10.1016/j.watres.2015.02.012>
7. Garduño-Jiménez, A.-L., Durán-Álvarez, J.-C., Ortori, C. A., Abdelrazig, S., Barrett, D. A., & Gomes, R. L. (2023). Delivering on sustainable development goals in wastewater reuse for agriculture: Initial prioritization of emerging pollutants in the Tula Valley, Mexico. *Water Research*, 238, 119903. <https://doi.org/10.1016/j.watres.2023.119903>
8. Han, Z., Jiang, J., Xia, J., Yan, C., & Cui, C. (2024). Occurrence and fate of microplastics from a water source to two different drinking water treatment plants in a megacity in eastern China.

- Environmental Pollution, 346, 123546. <https://doi.org/10.1016/j.envpol.2024.123546>
9. Kumar, A., & Boruah, B. M. (2014). Heparin assisted photochemical synthesis of gold nanoparticles and their performance as SERS substrates. *Journal of Nanoparticle Research*, 16, 2570. <https://doi.org/10.1007/s11051-014-2570-7>
  10. Nava, V., Frezzotti, M. L., & Leoni, B. (2021). Raman spectroscopy for the analysis of microplastics in aquatic systems. *Applied Spectroscopy*. <https://doi.org/10.1177/00037028211043119>
  11. Niu, J., Gao, B., Wu, W., Peng, W., & Xu, D. (2022). Occurrence, stability and source identification of small size microplastics in the Jiayan reservoir, China. *Science of the Total Environment*, 807, 150832. <https://doi.org/10.1016/j.scitotenv.2021.150832>
  12. Patra, K. B., & Baitharu, I. (2024). Assessment of microplastics and associated ecological risk in the Hirakud Reservoir, Odisha, India. *Journal of Water and Health*, 22, 1017–1032. <https://doi.org/10.2166/wh.2024.393>
  13. Pawak, V. S., Bhatt, V. K., Sabapathy, M., & Loganathan, V. A. (2024). Multifaceted analysis of microplastic pollution dynamics in the Yamuna River: Assessing anthropogenic impacts and ecological consequences. *Journal of Hazardous Materials*, 480, 135976. <https://doi.org/10.1016/j.jhazmat.2024.135976>
  14. Pérez-Guevara, F., Roy, P. D., Elizalde-Martínez, I., Kutralam-Muniasamy, G., & Shruti, V. C. (2022). Human exposure to microplastics from urban decentralized pay-to-fetch drinking-water refill kiosks. *Science of the Total Environment*, 848, 157722. <https://doi.org/10.1016/j.scitotenv.2022.157722>
  15. Perraki, M., Skliros, V., Mecaj, P., & Stamatis, G. (2024). Identification of microplastics using  $\mu$ -Raman spectroscopy in surface and groundwater bodies of SE Attica, Greece. *Water*, 16, 843. <https://doi.org/10.3390/w16060843>
  16. Phan, S., Ramtahal, J. J., & Luscombe, C. K. (2022). The effect of weathering environments on microplastic chemical identification with Raman and IR spectroscopy: Part I. Polyethylene and polypropylene. *Polymer Testing*, 116, 107752. <https://doi.org/10.1016/j.polymertesting.2022.107752>
  17. Phan, S., Ramtahal, J. J., & Luscombe, C. K. (2026). The effect of weathering environments on microplastic chemical identification with Raman and IR spectroscopy: Part II. Polystyrene, polyethylene terephthalate, and nylon 6. *Soft Matter*, 22, 3403–3419. <https://doi.org/10.1039/D5SM01102G>
  18. Piñon-Colin, T. J., Rodriguez-Jimenez, R., Rogel-Hernandez, E., Alvarez-Andrade, A., & Wakida, F. T. (2020). Microplastics in stormwater runoff in a semiarid region, Tijuana, Mexico. *Science of the Total Environment*, 704, 135411. <https://doi.org/10.1016/j.scitotenv.2019.135411>
  19. Prata, J. C., da Costa, J. P., Duarte, A. C., & Rocha-Santos, T. (2019). Methods for sampling and detection of microplastics in water and sediment: A critical review. *TrAC Trends in Analytical Chemistry*, 110, 150–159. <https://doi.org/10.1016/j.trac.2018.10.029>
  20. Prata, J. C., da Costa, J. P., Girão, A. V., Lopes, I., Duarte, A. C., & Rocha-Santos, T. (2019). Identifying a quick and efficient method of removing organic matter without damaging microplastic samples.

- Science of the Total Environment*, 686, 131–139. <https://doi.org/10.1016/j.scitotenv.2019.05.456>
21. Prosenc, F., Leban, P., Šunta, U., & Bavcon Kralj, M. (2021). Extraction and identification of a wide range of microplastic polymers in soil and compost. *Polymers*, 13, 4069. <https://doi.org/10.3390/polym13234069>
22. Ramírez-Álvarez, N., Rios Mendoza, L. M., Macías-Zamora, J. V., Oregel-Vázquez, L., Alvarez-Aguilar, A., Hernández-Guzmán, F. A., Sánchez-Osorio, J. L., Moore, C. J., Silva-Jiménez, H., & Navarro-Olache, L. F. (2020). Microplastics: Sources and distribution in surface waters and sediments of Todos Santos Bay, Mexico. *Science of the Total Environment*, 703, 134838. <https://doi.org/10.1016/j.scitotenv.2019.134838>
23. Range, D., Scherer, C., Stock, F., Ternes, T. A., & Hoffmann, T. O. (2023). Hydro-geomorphic perspectives on microplastic distribution in freshwater river systems: A critical review. *Water Research*, 245, 120567. <https://doi.org/10.1016/j.watres.2023.120567>
24. Secretaría de Medio Ambiente y Recursos Naturales. (2024, September 26). *Decreto por el que se declara zona de restauración ecológica el área de influencia de la presa Endhó, con una superficie total de 36,637-93-00 hectáreas, ubicada en el estado de Hidalgo [Decree Declaring the Area of Influence of the Endhó Dam, with a Total Area of 36,637-93-00 Hectares, Located in the State of Hidalgo, as an Ecological Restoration Zone]*. Diario Oficial de la Federación. <https://sidof.segob.gob.mx/notas/5739811>
25. Shen, M., Li, Y., Qin, L., Chen, X., Ao, T., Liang, X., Jin, K., Dou, Y., Li, J., & Duan, X. (2025). Distribution and risk assessment of microplastics in a source water reservoir, Central China. *Scientific Reports*, 15, 468. <https://doi.org/10.1038/s41598-024-84894-z>
26. Shruti, V. C., Jonathan, M. P., Rodríguez-Espinosa, P. F., & Rodríguez-González, F. (2019). Microplastics in freshwater sediments of Atoyac River basin, Puebla City, Mexico. *Science of the Total Environment*, 654, 154–163. <https://doi.org/10.1016/j.scitotenv.2018.11.054>
27. Tagg, A. S., Harrison, J. P., Ju-Nam, Y., Sapp, M., Bradley, E. L., Sinclair, C. J., & Ojeda, J. J. (2017). Fenton's reagent for the rapid and efficient isolation of microplastics from wastewater. *Chemical Communications*, 53, 372–375. <https://doi.org/10.1039/C6CC08798A>
28. Vayghan, A. H., Rasta, M., Zakeri, M., & Kelly, F. J. (2022). Spatial distribution of microplastics pollution in sediments and surface waters of the Aras River and reservoir: An international river in Northwestern Iran. *Science of the Total Environment*, 843, 156894. <https://doi.org/10.1016/j.scitotenv.2022.156894>
29. Wang, Z., Zhang, Y., Kang, S., Yang, L., Shi, H., Tripathee, L., & Gao, T. (2021). Research progresses of microplastic pollution in freshwater systems. *Science of the Total Environment*, 795, 148888. <https://doi.org/10.1016/j.scitotenv.2021.148888>

## Figures

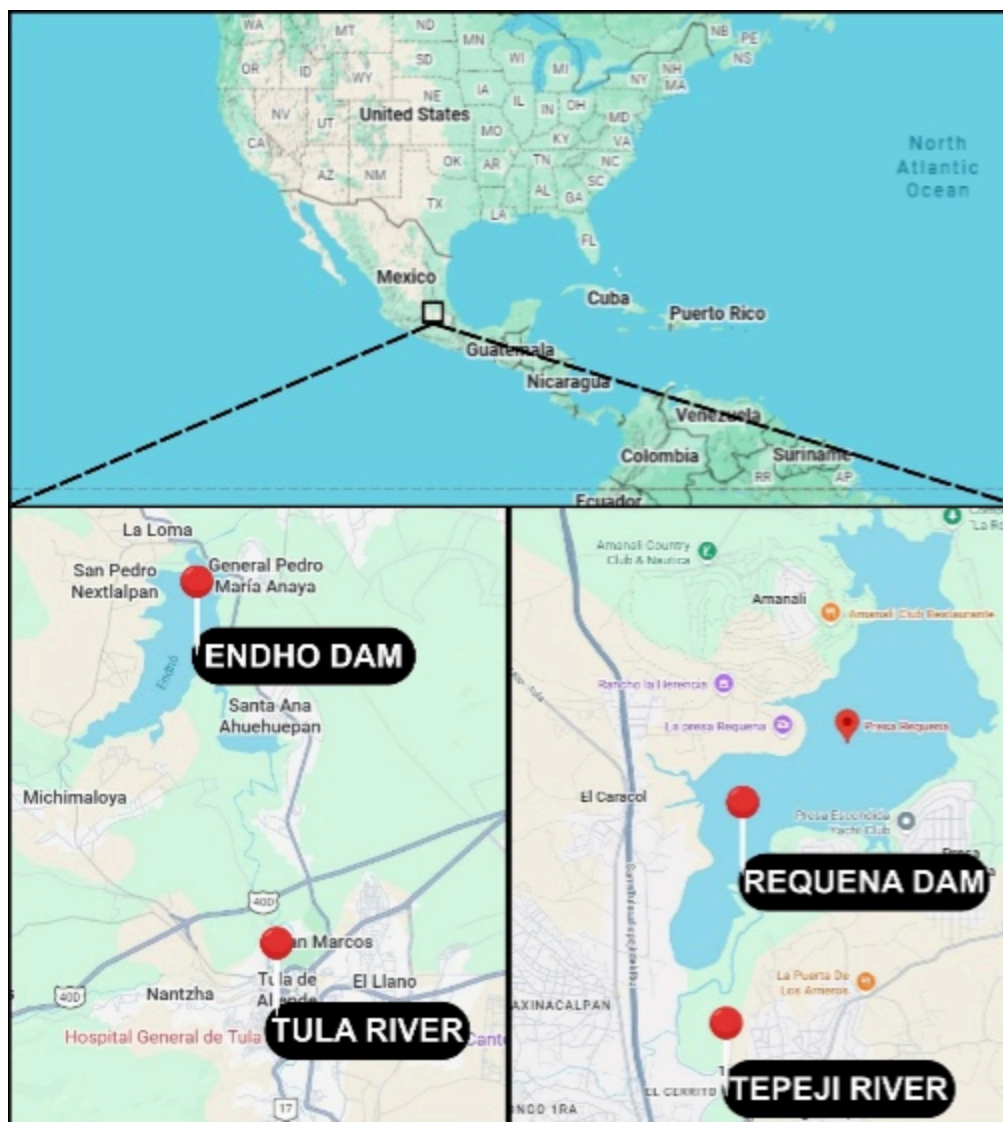


**Figure 1**

Location of the sampling sites in central Mexico: Tepeji River, Requena Reservoir, Tula River, and Endhó Reservoir, Hidalgo, Mexico

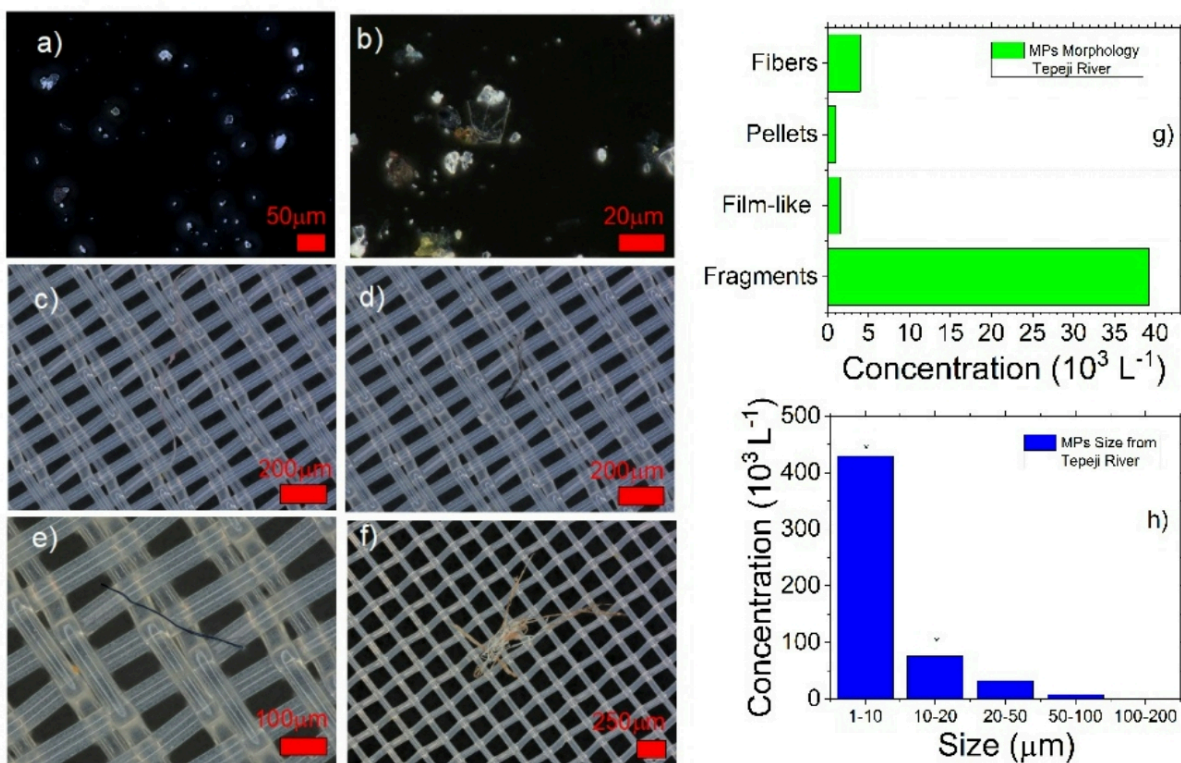


**Figure 2**

Representative microplastics observed in the Tepeji River and corresponding morphological and particle-size distributions. The asterisks in the size histogram indicate the two smallest size fractions, which are discussed separately as suspected MPs

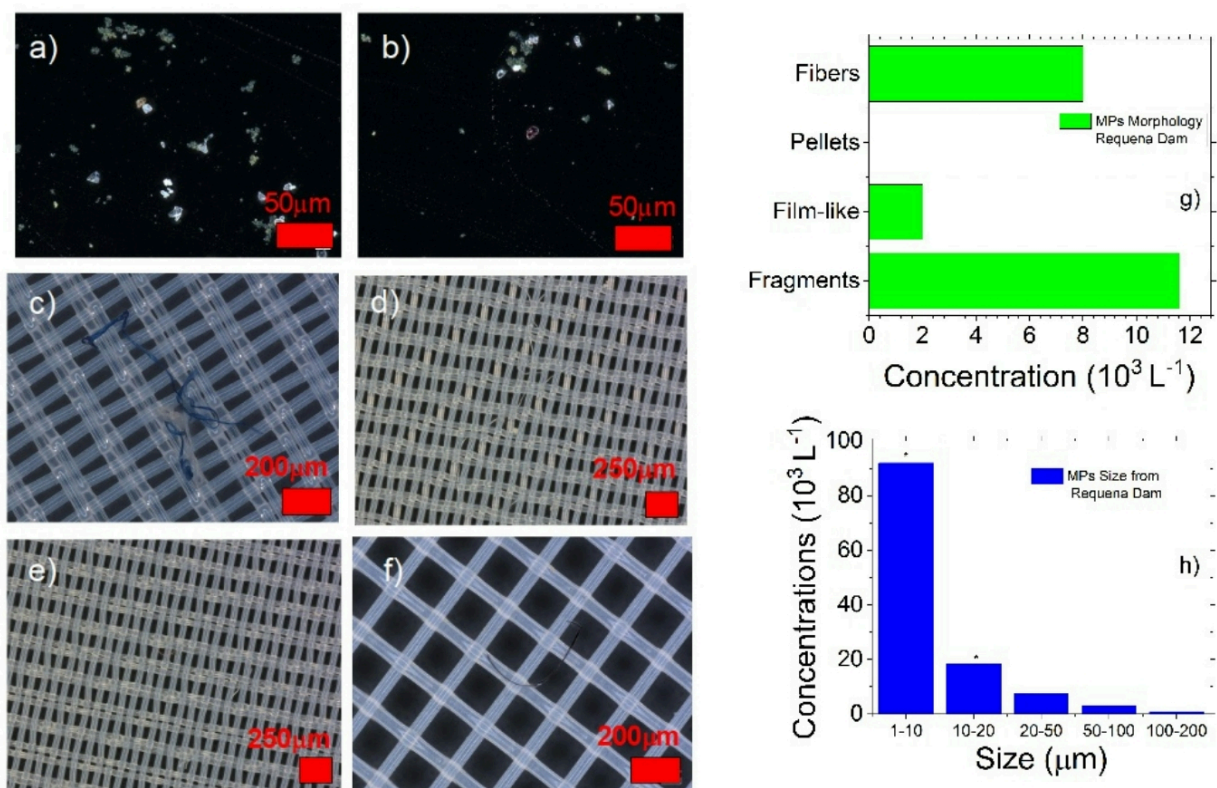


**Figure 3**

Representative microplastics observed in the Requena Reservoir and corresponding morphological and particle-size distributions. The asterisks in the size histogram indicate the two smallest size fractions, which are discussed separately as suspected MPs

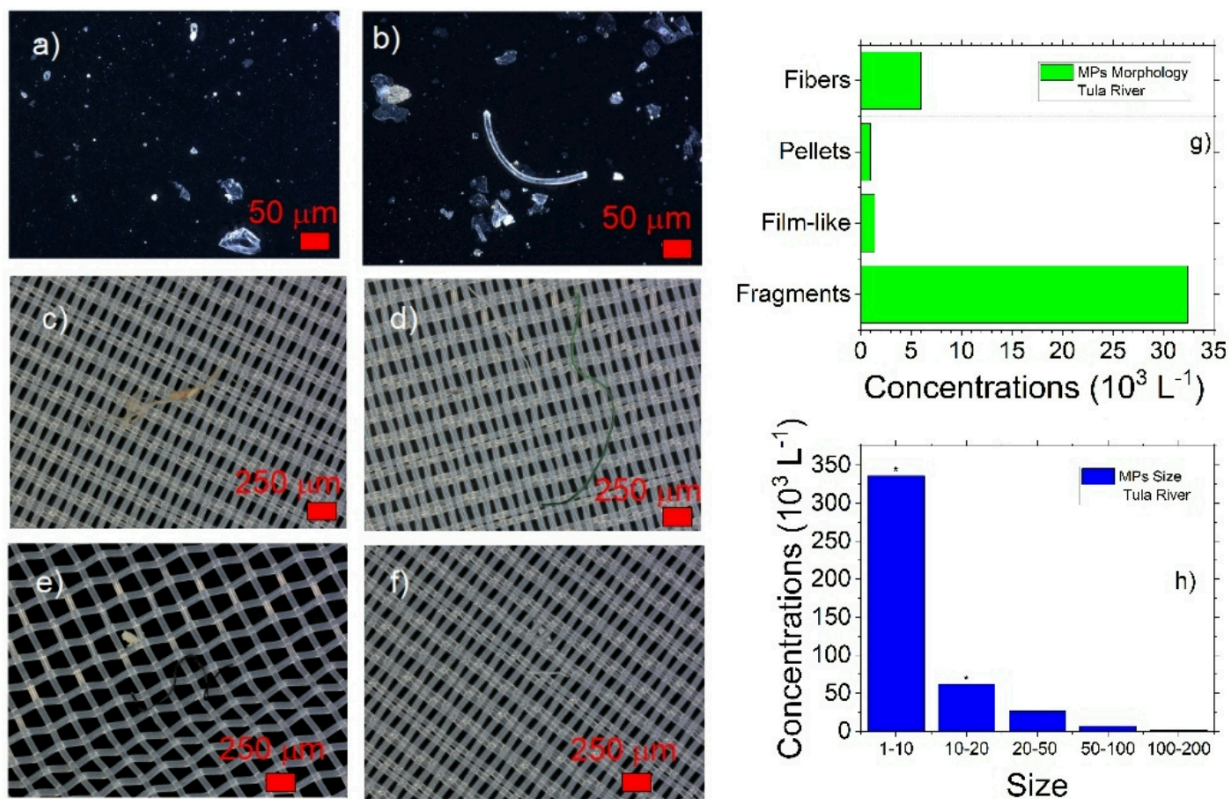


**Figure 4**

Representative microplastics observed in the Tula River and corresponding morphological and particle-size distributions. The asterisks in the size histogram indicate the two smallest size fractions, which are discussed separately as suspected MPs

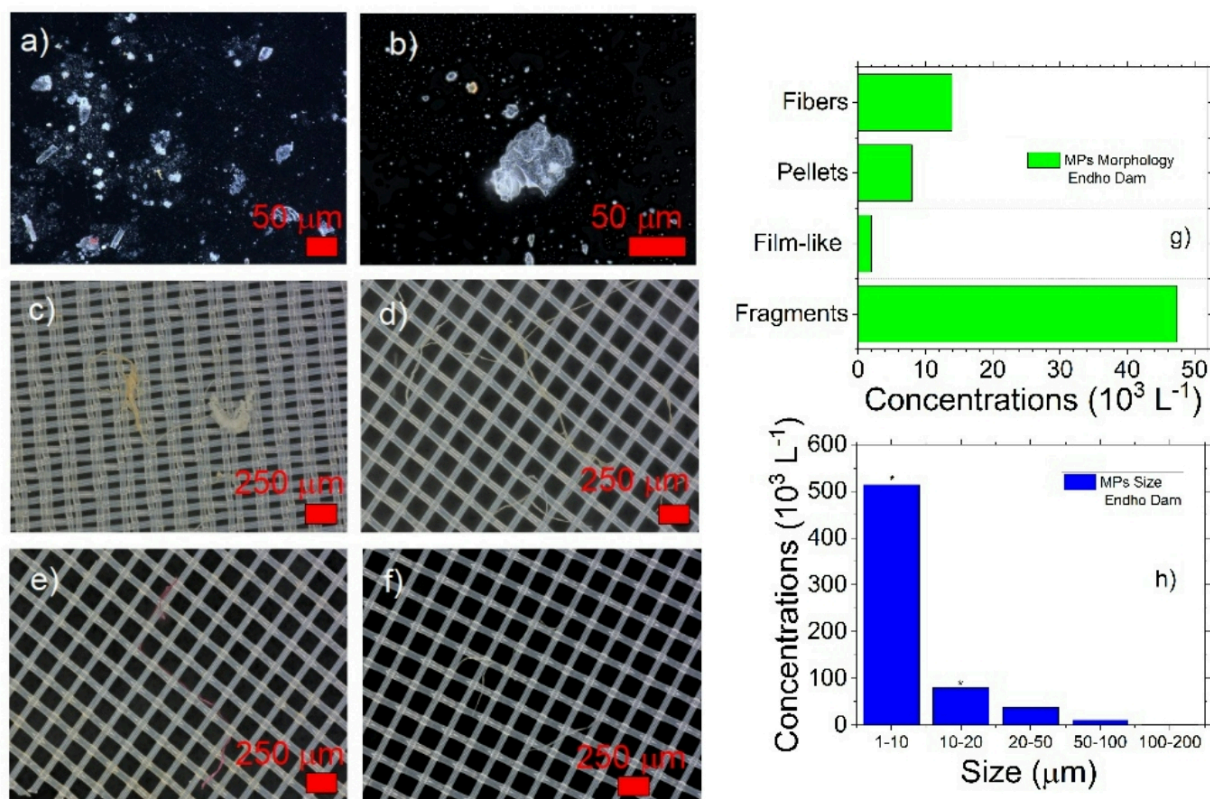


**Figure 5**

Representative microplastics observed in Endhó Reservoir and corresponding morphological and particle-size distributions. The asterisks in the size histogram indicate the two smallest size fractions, which are discussed separately as suspected MPs

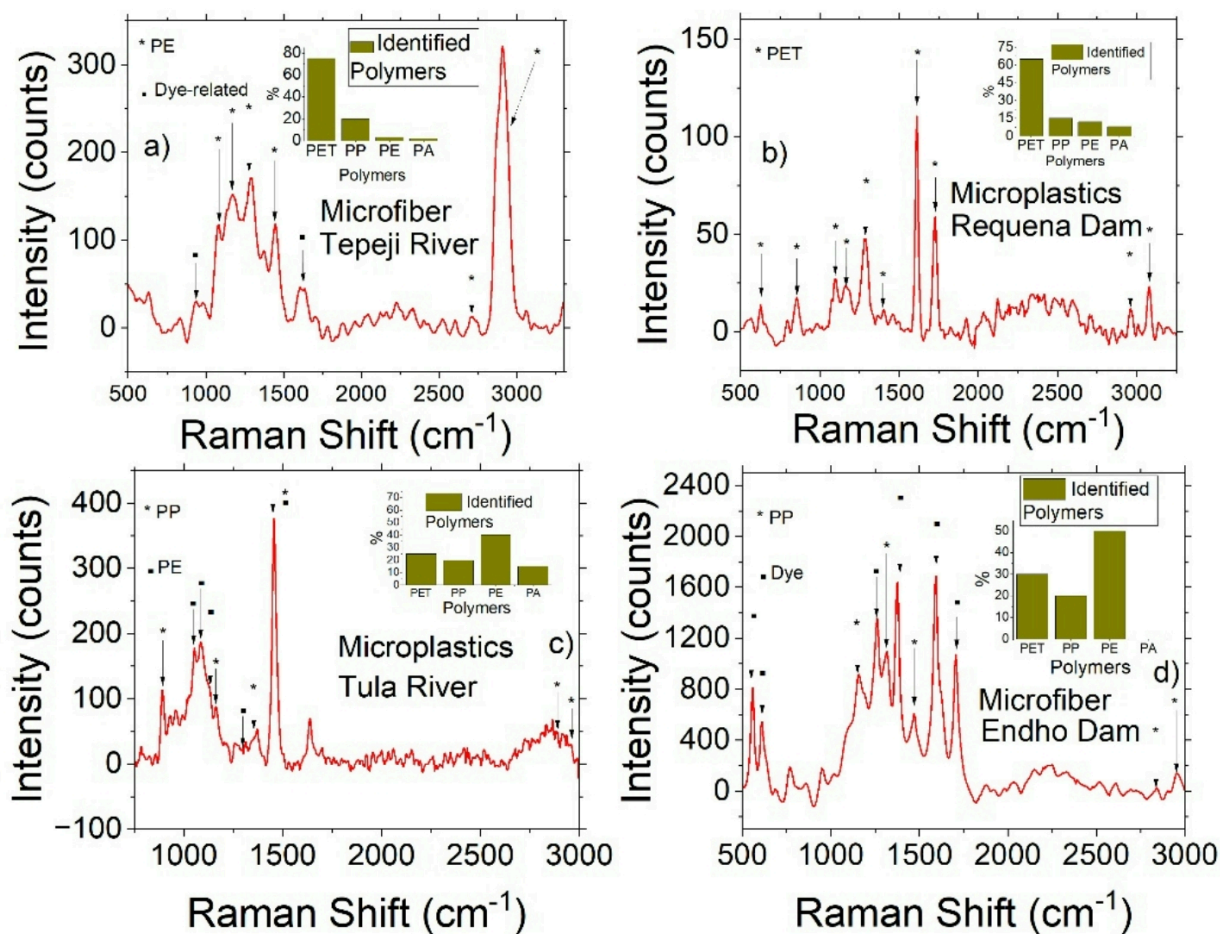


**Figure 6**

Representative Raman spectra of microplastics and microfibers. Asterisks mark polymer-related bands and squares indicate prominent dye-related contributions. Insets show the polymer distribution within the spectroscopically analyzed subset at each sampling site

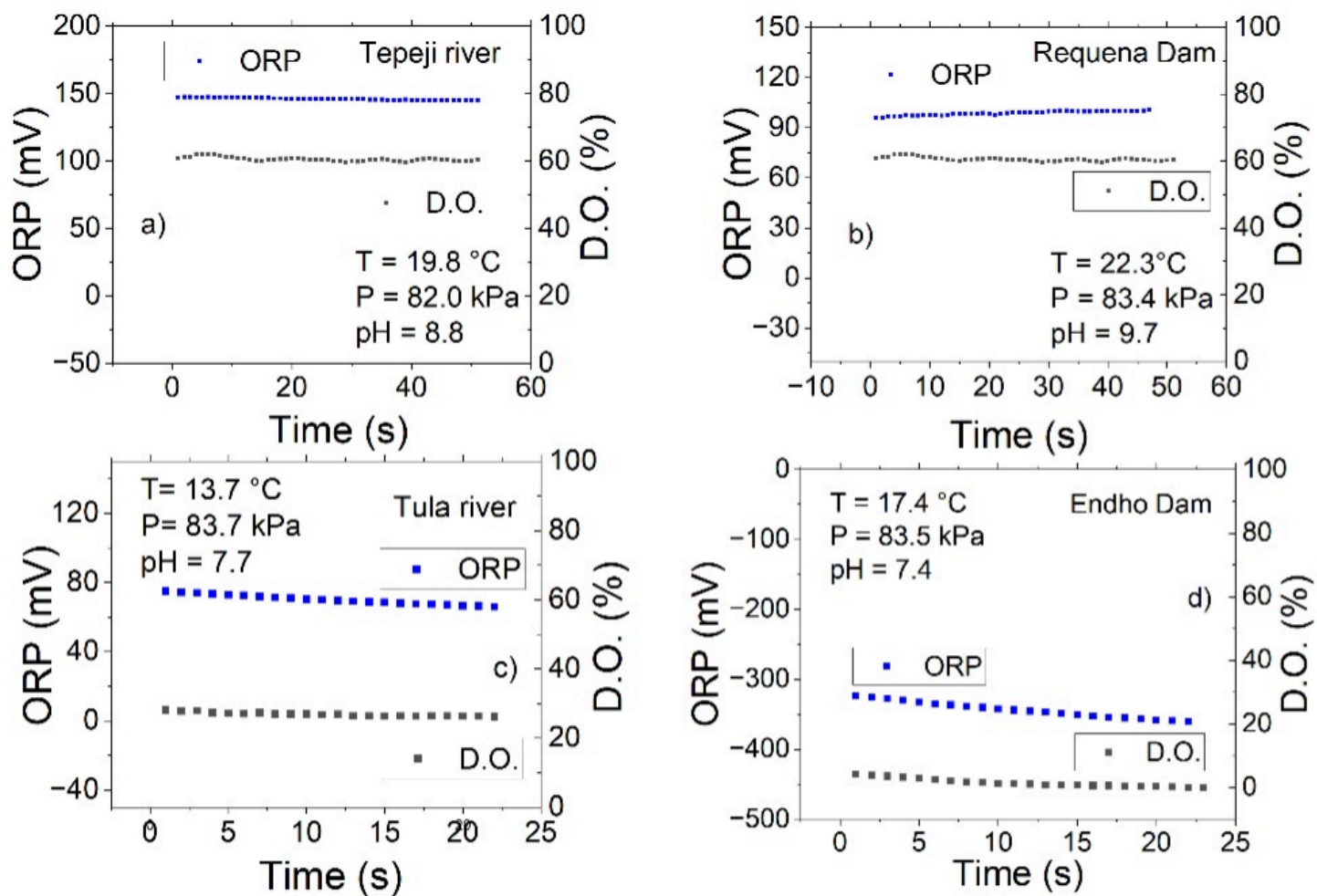


**Figure 7**

In situ oxidation–reduction potential (ORP) and dissolved oxygen (DO) measured during sampling at the (a) Tepeji River, (b) Requena Reservoir, (c) Tula River, and (d) Endhó Reservoir. Temperature, atmospheric pressure, and pH are indicated in each panel

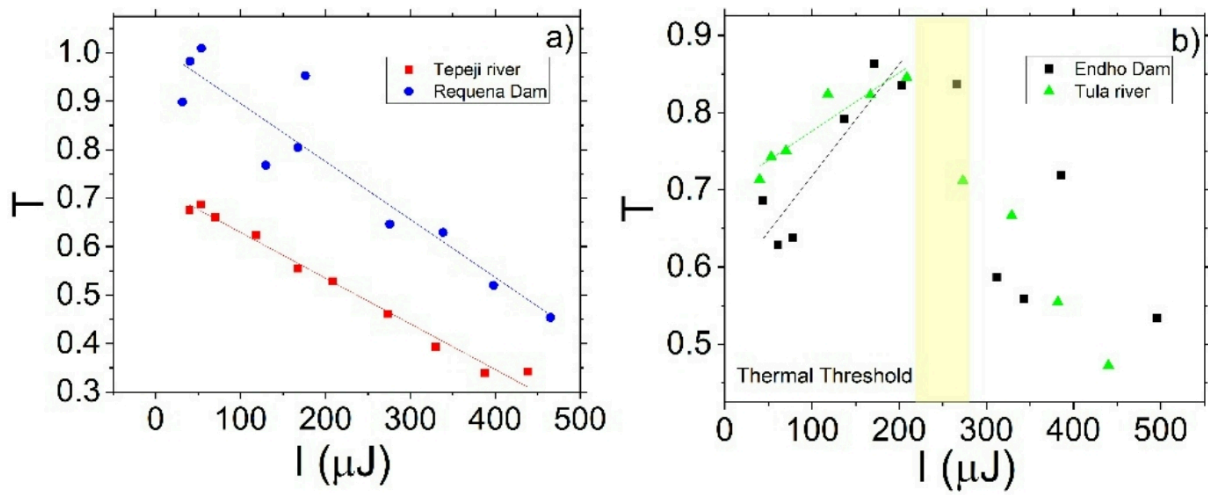


**Figure 8**

Transmission as a function of incident pulse energy at 532 nm for concentrated particulate suspensions from (a) the Tepeji River and Requena Reservoir and (b) the Tula River and Endhó Reservoir. The shaded region in (b) indicate the observed laser-induced thermal/photothermal threshold