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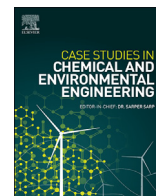
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A new method for microplastic sampling and isolation in mountain glaciers: A case study of one antisana glacier, Ecuadorian Andes



Marcela Cabrera^b, Bryan G. Valencia^{a,*}, Oscar Lucas-Solis^a, Jaqueline L. Calero^a, Luis Maisincho^{c,d}, Bruno Conicelli^a, Gabriel Massaine Moulatlet^a, Mariana V. Capparelli^a

^a Facultad de Ciencias de La Tierra y Agua, Universidad Regional Amazónica Ikiám, Km 7, Vía Muyuna, Tena, Napo, Ecuador

^b Laboratorio Nacional de Referencia Del Agua, Universidad Regional Amazónica Ikiám, Ecuador

^c Instituto Nacional de Meteorología e Hidrología (INAMHI), Iñaquito, N36-14 y, Corea, Quito, Ecuador

^d Institut des Géosciences de L'Environnement (IGE, UMR 5001), 38000, Grenoble, France

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ABSTRACT

Microplastic contamination has become ubiquitous in terrestrial and marine environments. Recent studies have shown that the wind can transport and deposit microplastics in high mountain ecosystems, but microplastic contamination therein is unknown. Because mountain glaciers are the primary source of drinking water for large urban areas in the Andes, assessing recent and historical microplastic contamination is crucial. Surface snow can indicate recent microplastic deposition, whereas glacial ice cores can provide information on historical contamination. At mountain glaciers, the inhospitable conditions and the difficult accessibility are limiting factors for sampling. Therefore, sampling and laboratory analytical methods have to be integrated and planned ensuring replicability. Here, we present 1) a new methodology to identify sampling areas within the accumulation zone of a glacier to obtain samples of surface snow and ice cores; 2) a less-manipulative analytical technique for the preparation and isolation of microplastics derived from glaciers. In addition, we identified the minimum amount of sample necessary to obtain robust data on contamination by microplastics.

1. Introduction

Microplastic (hereafter, MP) contamination has been found across several localities. A wide range of studies in marine and terrestrial environments have demonstrated MP contamination near populated areas [1]. However, studies reporting MP contamination in remote areas, such as high mountains, are uncommon. While MP contamination near urban areas is due to multiple sources, in remote areas, atmospheric transport is the most likely contaminant carrier [2,3]. Glaciers of high mountains represent a research gap of worldwide MP distribution and may act as contaminant sinks [3,4]. For instance, ice cores have been collected from ice sheets and glaciers to assess MP contamination in the Arctic and Antarctic, but not yet in the high-altitude tropical glaciers [5–7]. Countless windborne particles of natural and anthropogenic origin such as MP can be continuously deposited and trapped in the snow, becoming part of glaciers that are the primary source of drinking water for urban areas [2,8,9]. Consequently, assessing the degree of MP contamination in glacial ecosystems is crucial.

It is well known that MP can be transported by water runoff, reaching

rivers and marine environments. Recently, some studies have suggested the plausibility of MP as windborne, enabling its accumulation on mountain glaciers [2,4,10]. Due to glacier melting, MP that have accumulated because of atmospheric deposition could be released into watercourses and reach natural urban water reservoirs [4]. However, the potential increase in MP contamination caused by glacier melting remains uncertain. Therefore, it becomes relevant implementing standardized analytical protocols for MP sampling and processing in surface snow and ice cores [3]. Current methodologies used for the collection and analysis of wind deposited MP are diverse and not standardized [11]. To our knowledge, there are no methodologies on MP contamination and isolation tailored explicitly for snow and glacier ice. Studies carried out on high elevation glaciers in the Alps and Tibetan Plateau [4,8], used methodologies developed for general MP assessments or to test specific hypotheses, such as the air transportation of MP via wet snow. The protocols for collecting surface snow in glaciers consist of obtaining thin layers in random locations, but only within the ablation zone or at the base of the glacier accumulation zone [8]. Uneven sampling along the elevation gradient of the glacial may be insufficient to understand

* Corresponding author.

E-mail address: bryan.valencia@ikiam.edu.ec (B.G. Valencia).

transport dynamics for MP contamination. Then, consolidated ice and surface snow retrieved along an elevational gradient can provide a comprehensive spatiotemporal depiction of MP contamination on a glacier. While surface snow can indicate a recent deposition, glacial ice cores can provide information on historical contamination.

Here, we present 1) a new methodology to define the priority sampling areas along an elevation gradient of a mountain glacier, to obtain surface snow samples or glacier ice core and 2) a less-manipulative and cost-effective methodology for preparation and quantification of MP from glacier-derived samples. The methodology allows sampling planning in remote areas where low MP contamination is expected and where collecting large amounts of material is difficult. This study demonstrates the applicability of our methodology in identifying and monitoring MP contamination in surface snow and ice core samples that come from the remote Antisana Glacier system, in the tropical Andes of Northern Ecuador.

2. Methods

2.1. Study area

Expeditions to the Glacier 15- α (-0.478693° , -78.147966°) of the Antisana volcano took place between December of 2019 and February of 2020. The Antisana Volcano (Fig. 1) supports the largest and the fourth-highest ice cap of the Ecuadorian Andes (total surface area of ~ 15 km² and 5753 m above sea level; hereafter, masl). The Glacier 15- α was selected because it is the most studied and monitored outlet glacier on the Antisana ice cap [12]. Precipitation is bimodal, with enhanced

rainfall from February to June and from September to November. The mean temperature at the proglacial margin (4850 masl) ranges between 0°C and 2°C [12]. Few and small populated areas lie around the Antisana ice cap. However, Quito, the largest city in Ecuador (~ 2.8 million inhabitants), is ~ 50 km away from Antisana (Fig. 1B). The Antisana glaciers are an important water source for Quito city and their runoff also supports the paramo wetlands, an essential highland ecosystem that provides several ecosystem services [9].

2.2. Field sampling and data collection

Mountain glaciers are remote areas of difficult access. Understanding the full picture of MP contamination depends on careful sampling-planning and the identification of collection sites. Remote sensing data enables the identification of ascent routes taken by mountaineering crews to prioritize site selection based on the accessibility.

The surface sampling sites were selected using the overlap between elevation contours generated every 100 m, a terrain slope, and a summit path. The spatial analyses were performed with the freeware software QGIS 3.10 and plugin-tools from GRASS and SAGA (Fig. 2). The 100 m elevation interval represents the maximum sampling resolution that could be attained in the field. In February 2020 we collected a set of five surface snow samples, in triplicate, every 100 m from 5100 to 5600 m and an 8-m ice core was drilled at 5520 m, as summit (5740 m) could not be reached carrying all the ice coring gear. The minimum sampling elevation (5100 m) corresponded to the equilibrium line altitude (ELA) range for the Antisana glacier [13]. Areas far below the ELA were excluded as they may experience MP enrichment due to snow ablation.

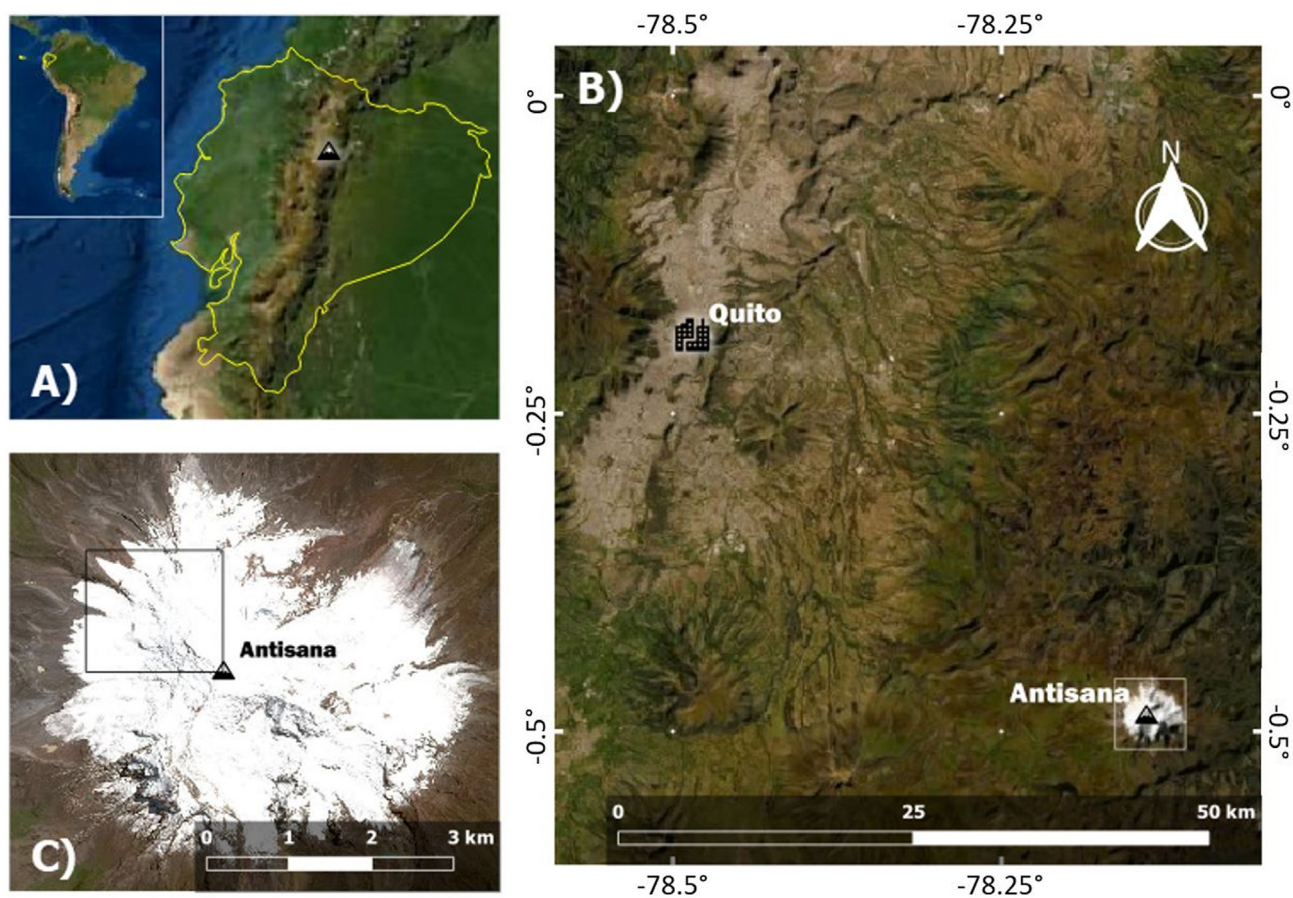


Fig. 1. Map of the study area. A) Location of the Antisana volcano, in the Napo province of Ecuador. B) The Antisana is located about 50 Km away from the city of Quito, the capital of Ecuador, and the largest urban area of tropical Andes. C) Detailed image of the Antisana volcano showing the summit path on the glacier 15- α in the northwestern region of the volcanic cone. Background satellite imagery was derived from free Yandex and Esri Imagery sources.

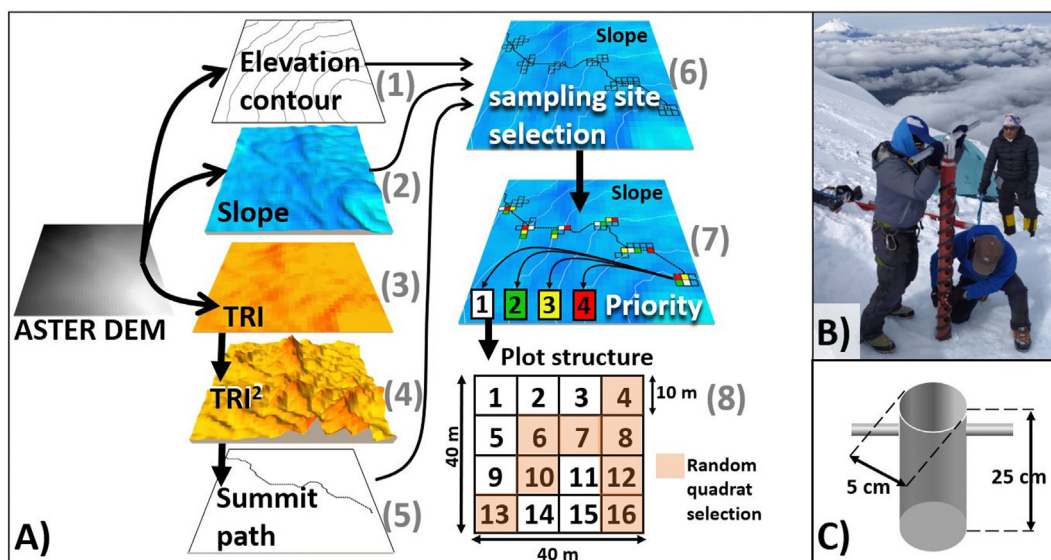


Fig. 2. Depiction of the spatial analysis conceptual-diagram (A), ice core sampler (B), and surface snow sampler (C). The elevation contours (1), slope (2), and TRI (3) were directly derived from an ASTER DEM (30-m grid). The summit path (5) was generated using squared TRI values [TRI², (4)]. Sampling sites [black open squares (6)] were selected intersecting the elevation contours (1), slope (2), and summit path layers (5). The sampling sites [black open squares on (6)] were ranked from low to high slope values. The highest priority to lay down sampling plots (8) was assigned to four sites with the lowest slopes from one (highest priority; white cells) to four (low priority; red cells). Sampling plots (h; 40 m × 40 m) were subdivided into sixteen 10-m quadrats. Eight quadrats were selected randomly and a sample was taken in the center of each chosen quadrat. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

The elevation contours and terrain slope were generated from an ASTER elevation model (Advanced Spaceborne Thermal Emission and Reflection Radiometer, GDEM 003; 30-m resolution; hereafter, ASTER DEM). The aim was to identify areas at the selected sampling elevations with similar terrain slopes, i.e., similar snow and MP accumulation. Areas with high terrain slopes may be susceptible to slumping and were avoided. A least-cost-path analysis was derived from a squared TRI (Topographic Ruggedness Index) to identify the most accessible summit path and ensure the chosen sampling sites were reachable. TRI values estimate the elevational difference of a central cell against the neighboring cells. Because TRI values increase proportionally to the elevational differences between the central and neighboring cells, cells with low TRI values represent areas with little topographic irregularities. Therefore, a least-cost-path analysis run on a squared TRI further avoids areas with topographic irregularities when generating a summit path. A preliminary survey was carried out ahead of the actual field expedition to ensure its feasibility.

To ensure the lowest slope was selected, the slope cells within 15 m from the contour line, and no further than three cells from the summit path were selected and ranked from low to high values. Four cells with the lowest slope values were retained as suitable sampling areas where one represented the top priority and four the lowest.

Within the selected sampling sites, the surface snow samples were retrieved within 40 m by 40 m plots. Each plot was divided into 16 quadrants from which half were selected using random numbers generated in R software (R version 3.4.3). A metal sampler with known dimensions ($\varnothing = 5$ cm, height = 25 cm, Fig. 2C, and Table 1) was used to retrieve a consistent snow volume (450 mL) at the center of each chosen quadrant. While taking the surface snow samples, the mountaineering crew, trained to avoid MP contamination, remained downwind to the sampling points. Surface snow samples were stored in 33 cm by 24 cm sealed and pre-cleaned plastic bags to avoid MP contamination. Samples were transported in coolers filled with dry ice to avoid melting. The samples were stored in a freezer at -18 °C and analyzed at the National Water Reference Laboratory (NWRL) housed at Universidad Regional Amazónica Ikiam.

The pre-cleaning of the plastic bags for sample collection consisted of

washing the bags three times with Milli-Q water (18 M Ω cm) and drying at room temperature. Then, the bags selected as blanks were filled with Milli-Q water once again and were frozen at -18 °C, filtered and had MP quantified. The MP contamination of blank samples resulted in less than 5% of the contamination found in the snow and ice core samples. Whenever possible, plastic containers should be avoided to store samples for microplastic analysis. However, for this study, collecting the samples in glass or metal containers was restrictive due to weight limitations.

2.3. Standardization of methodology for detecting MP

The MP laboratory analysis consists of two steps: (i) extraction-purification, and (ii) isolation and identification. To validate our methodology, we selected three random samples from a suite of surface snow samples collected at different altitudes and three random samples from the ice core. Our methodology was based on an adapted version of the method used in other glaciers to analyze surface snow [2,8,10] and samples from the ice cores [5].

Before the MP analysis, Total Organic Carbon (TOC) was determined using a total organic carbon analyzer (TOC-L Shimadzu, Japan) for snow and ice core samples. Samples must be chemically treated to remove the organic content when it is high. For instance, our samples contained a negligible amount of organic matter (TOC 0.5 mg/L, which is similar to pure water) and therefore, organic matter and debris dissolvers, such as hydrogen peroxide (H₂O₂) were avoided to minimize sample manipulation.

Before starting with the extraction of MP, we promoted two recovery experiments in triplicate using high-density polyethylene red (range size: <250 μ m and >65 μ m; density = 940 kg/m³). Ten MP were deposited into 250 ml clean Milli-Q water and subjected to the entire process detailed below with centrifugation conditions of 3000 and 4000 rpm applied for 5 min each. Thereafter, the filters were stored in capped glass Petri dishes for further visual identification.

The average MP recovery in the two experiments were $86\% \pm 5.7\%$ (centrifugation at 3000 rpm; with three replicates had an 8.6 mean and 0.6 standard deviation) and $153\% \pm 15\%$ (centrifugation at 4000 rpm, with three replicates had a mean of 15 and 1.5 standard deviation). The

Table 1
Summary of methodologies implemented in various studies that analyzed MP contamination in either surface snow or ice core samples.

| surface snow samples | | | | | | | | |
|--|-----------------|--|---|---|--|--|--|------------|
| Location | Altitude (masl) | Sample type | Filtration | Separation | Organic Matter removing | MP Abundance | MP Colors | References |
| Ablation tongue of Forni Glacier, Italian Alps, Europe | 2580 | Cryonite and supraglacial debris | Water-jet pump with glass fiber filters ($\varnothing = 47$ mm; pore size, 0.45 μm) | Saturated sodium chloride solution (365g/L-1) | Hydrogen peroxide (H_2O_2) solution (15%) | 74.4 items kg ⁻¹ of sediments (dry weight): Fibers 65.2%; Fragments 34.8% | Black 31% Blue 22% Red 17% Transparent 17% Light blue 9% Violet 4% | [8] |
| Europe (Swiss Alps, North Germany) and Artic Pole | N/A | Wet snow deposition | Aluminum oxide filters ($\varnothing = 25$ mm; pore size, 0.2 μm) | N/A | N/A | In Europe: 24.6 $\pm 18.6 \times 10^3$ items L-1; Fibers 1.431 $\pm 0.325 \times 10^3$ items L-1 In Arctic: 1.76 $\pm 1.58 \times 10^3$ items L-1; Fibers 1.38 $\pm 1.10 \times 10^3$ items L-1 | N/A | [2] |
| Antisana Glacier, Ecuadorian Andes, South America | 5753 | Surface Snow | Borosilicate laboratory glass vacuum filtration equipment with cellulose nitrate filters ($\varnothing = 47$ mm; pore size, 0.45 μm) | 20 mL of NaCl (1200 kg/m ³) and centrifugation (3000 rpm) | N/A | Mean of 101.2 items L-1; Fibers 71.5%; Fragments 7.8%; Other 20.7% | Transparent 51% Blue 30% White 9% Red 6% Cream 3% Other 1% | This study |
| Ice core | | | | | | | | |
| Location | Altitude | Sampling | Filtration | Separation | Organic Matter removing | MP Abundance | MP Colors | References |
| Arctic Ocean | 0 | Drilling with an attached motor and a stainless-steel core barrel of 12.5 cm diameter; transportation with clean Ziploc bags (polyethylene). | Büchner funnel and a vacuum flask with glass microfiber paper ($\varnothing = 47$ mm; pore size, 1.2 μm). | N/A | N/A | Ice cores: 2–17 items L-1, Fragments and Fibers | Ice Cores Blue 53% Red 10% Pink 9% Yellow 7% Black 5% Green 3.5% Transparent 3.5% White 3% Grey 3% Orange, Purple and brown 3% | [7] |
| Antarctic sea ice | 0 | Drilling with an electro-polished stainless-steel corer. Transportation with triple-bagged in acid-cleaned PE plastic bags. | Silver membrane filter ($\varnothing = 25$ mm; pore size, 5 μm) Aluminum oxide filters ($\varnothing = 25$ mm; pore size, 0.2 μm). | N/A | Before filtration 10 mL H_2O_2 (35%) per 100 mL. After filtration, 40 mL of H_2O_2 . | Mean of 20.38 items L-1. | N/A | [6] |
| Arctic sea ice | 0 | Various sources of 1–3.5 m sea ice cores. | Millipore nitrocellulose membranes (pore size, 0.22 μm). | N/A | N/A | 3.8×10^{-2} - 2.3×10^{-1} items L-1 | Mostly blue, black, green and red. | [19] |
| Arctic sea ice | 0 | Drilling with a Kovacs 9 cm diameter corer; Transportation into plastic bags (polyethylene tube films (LDPE)). | Aluminum oxide filters ($\varnothing = 47$ mm; pore size, 0.2 μm). | N/A | 40 mL H_2O_2 (35%). | Range from 1.1 $\pm 0.8 \times 10^3$ items L-1 to 4.1 $\pm 2.0 \times 10^3$ items L-1 | N/A | [5] |
| Antisana Glacier, Ecuadorian Andes, South America | 5753 | Drilling with a Pico Hand Augers metal sampler ($\varnothing = 5$ cm, height = 25 cm) (Fig. 2) | Borosilicate laboratory glass vacuum filtration equipment with cellulose nitrate filters ($\varnothing = 47$ mm; pore size, 0.45 μm) | 20 mL of NaCl (1200 kg/m ³) and centrifugation (3000 rpm) | N/A | Mean of 69.2 items L-1; Fibers 66.5%; Fragments 8.9%; Other 24.7% | Transparent 45% Blue 32% Red 14% White 8% Yellow 1% | This study |

recovery rate of MP in the 4000 rpm experiment resulted in plastic deformation and particle fracturing that explains percentages above 100%. Our results are consistent with Elkhatib and Oyanedel-Craver, who reported that centrifugation ranging from 4000 to 4500 rpm applied from 2 to 20 min intervals might cause deformation, compression, or breaking of the plastic particles, resulting in an inaccurate determination of the MP abundance [14]. The MP loss associated with plastic particles adhering to the filtration funnel was about 20% when

centrifuging at 3000 rpm, resulting in neither particle fracturing nor deformation.

2.4. Microplastic extraction/purification

Based on the recovery experiments, MP processing started by melting both ice core, surface snow and blank samples at room temperature. The ice core was cut with an electric saw in sections of 10 cm and each section

was washed with Milli-Q water to remove adhered particles. Subsequently, for both sample types, a volume of 250 ml was filtered through 0.45 μm cellulose nitrate membranes with $\varnothing = 47$ mm (Pall Corporation), using a borosilicate laboratory glass vacuum filtration equipment. The walls of the filter cup were rinsed with Milli-Q to avoid loss of MP adhered to the filter cup. The membrane with the filtrate was rinsed into a graduated precipitate cylinder with 20 mL of Sodium Chloride (NaCl, 1200 kg/m^3) solution using a filter of 0.22 μm pore size (Millipore Whatman).

The samples were passed through 15 ml centrifuge tubes, previously cleaned with ultra-pure water, and centrifuged at 3000 rpm for 5 min to remove sediment particles. The supernatant was filtered with 0.45 μm cellulose nitrate membranes of $\varnothing = 47$ mm for later visual inspection.

To mitigate MP contamination during the processing of the samples, only clean cotton lab coats were worn by the laboratory crew, all working surfaces were wiped with ethanol and the glass filtration equipment and glass Petri were rinsed with Milli-Q before usage. The Milli-Q water used in some phases of the methodology was previously filtered to avoid MP contamination.

2.5. Microplastic isolation

Filters were examined under an optical microscope (Motic BA 210E, 10X/0.25 zoom) by an experienced observer in the identification of MP. All MP particles were counted and classified into categories according to their shape (plastic fragments, sphere, fibers, and plastic films) [15] and color. Only MP > 60 μm were detected in this study. To be classified as MP, the fibers had to be equally thick through their entire length and should not be entirely straight [16]. Normally, aged plastic, such as expected in environmental samples, present embrittled and weathered surfaces and have irregular shapes with broken and sharp edges [17]. Also, MP might present clear and homogeneous colors. Commonly, the color could be a plastic-type identifier that tend to vary from transparent to variations of white, bright orange, blue, green, purple, and black [3, 10]. To reduce the possible error due to cross-contamination in the samples, the MP mean abundance of the three blanks was used to correct all the results.

3. Results and discussion

3.1. Microplastic isolation

Three samples of surface snow and ice core were analyzed to test the effectiveness of our methodology. In 250 ml analysis for surface snow, a total of 270 MP particles were found (an average of 89.7 MP per sample). In the same volume, we found a total of 158 MP particles (an average of 52 MP per sample) in ice core samples. The majority of visible particles included fibers, films, fragments, and spheres. Fibers (71% surface snow - 66% ice core) were the most abundant shapes in samples. The size of MP ranged between 60 and 2500 μm .

On average, transparent (51%), blue (30%), white (9%), and red MP (6%) were the most abundant colors in the surface snow samples. Moreover, a small number of cream and yellow MP were observed. Transparent (45%), blue (32%) and red (14%), and white MP (8%) were the most abundant colors in the ice core samples.

The MP cross-contamination was negligible since the abundance in blank samples was $<5\%$ of the abundance of MP detected in any of the field samples. The abundance of MP found in our samples was substantial to be considered as accidentally deposited during expeditions or other studies done at the Antisana Glacier 15- α . A comparison with MP abundances found in other glacial environments can be found in (Table 1). Thus, we hypothesized that MP are being transported from multiple sources and have deposited in the glacier over time.

3.2. Comparison between different methodologies

MP studies in glaciers are limited (Table 1), so methodologies to detect MP from atmospheric sources are taken as references, assuming this is the main source of MP entry to remote areas [8,10]. Overall, the protocols can be divided into the following stages: sampling, pre-treatment, density separation, post-treatment, and analysis [11]. However, the methodologies used for the sampling, treatment, and analysis of atmospheric MP are diverse and not standardized.

To sample MP in high mountain areas, rainfall samplers or particle fallout collectors have been used [10]. However, on high altitude glaciers where the weather and glacier surface are ever changing, rainfall and particle fallout monitoring is generally unfeasible [3]. Alternatively (Ambrosini et al., 2019 [8]), collected cryoconites and supraglacial debris, and were the first reporting MP contamination on a high-altitude glacier, at the ablation zone of the Italian Alps [8]. Cryoconites are formed at the ice-atmosphere interface or in the presence of meltwater and can accumulate and concentrate anthropogenic contaminants, such as MP [18]. Both Cryoconites and supraglacial debris are only found at the ablation surface of glaciers. At the ablation zone, the surface of the glacier melts and sublimates, enhancing the concentration of contaminants such as MP [8] in comparison with surface snow samples collected from higher elevation parts of glacial environments. For instance, the high MP contamination found by (Ambrosini et al., 2019 [8]) and (Bergmann et al., 2019 [2]) in sediments and surface snow samples from ablation zones of the Swiss Alps, are not directly comparable to MP contamination found in this study [2,8].

We report high abundances of MP in ice core samples. Ice cores have been collected to quantify the MP contamination of the Arctic and Antarctic low elevation glacial environments [5–7], as these ecosystems are considered important global MP sinks. High-altitude tropical glaciers are still data gaps and the question if MP accumulates in mountain ice cores, as it does in low elevation glaciers, is still to be answered. For that, specific methodologies to quantify MP in mountains are required (Table 1) [19]. Fresh and saltwater samples have similar processes for sampling and analysis, but there are differences in the MP distribution in each system due to several factors, such as water density and salinity [20]. Therefore, as many environmental and biological factors may affect the vertical distribution of MP [5], no consistent patterns have been found in the vertical distribution of MP from sea ice cores so far [6,7,19]. The likely explanation is that low elevation glaciers are subject to sea ice dynamics to which tropical mountain glaciers are not [7]. Thus, tropical glaciers ice cores may be used as stratigraphic indicators of atmospheric MP deposition providing valuable information on historical contamination [4].

Until now, surface snow sampling has consisted of the use of non-plastic (e.g. steel or glass) and unstandardized collectors, such as mugs, spoons, or soup ladles [2]. In contrast, we have used metallic devices (Fig. 2), which allowed us to obtain samples with similar volumes and to avoid excessive manipulation. In mountain glaciers, due to the difficulty of carrying the sampling equipment and the samples, lighter materials made of plastic instead of steel or glass containers are recommended (Table 1). To minimize MP sample contamination, non-plastic materials must be used and the sampling plastic bags must be pre-cleaned. Furthermore, blanks must be used to assess cross-contamination [2,5–7].

In the laboratory the ice and surface snow samples must be sectioned and melted. The sample sectioning may vary depending on the aim of each study. Here, we decided the length cut of 10 cm, in accordance with (Kanhai et al., 2020 [7]) and (Peeken et al., 2018 [5]), but the diameter of the collected ice cores in other studies ranged from 5 cm to 12.5 cm (Table 1) depending on the ice corer gear. In glacial environments the use of small diameters (~ 5 cm) and light ice coring gear has proven effective to collect samples and detect MP contamination.

The main pre-treatment applied to concentrate MP particles from ice

and surface snow samples was filtration. Due to the low amount of debris and organic matter found in surface snow samples compared to wastewater [2,21]. Glass fiber, aluminum oxide, silver membrane, and cellulose nitrate filters have been used to filtrate both surface snow and ice cores. We used and recommended cellulose nitrate filters, as they are inexpensive and easy to acquire. A pore size $>0.2\ \mu\text{m}$ avoids clogging and filtering of particles that interfere with the analysis, as particles $<60\ \mu\text{m}$ are not identifiable under the microscope.

After the filtration, density separation is required to separate MP from other particles. Concentrating MP allows their quantification and characterization [20]. Although zinc chloride (ZnCl_2) solutions are common for the density separation in atmospheric MP samples, salts such as NaCl, CaCl_2 , ZnBr_2 , NaBr and NaI can also be used [11,21]. A saturated NaCl solution ($1200\ \text{kg/m}^3$) was used for density separation, as indicated by (Ambrosini et al., 2019 [8]), because it has a low toxicity and is cost effective [8]. For instance, the saturated NaCl solution is suitable to float polymers such as polyethylene, polypropylene, polyamide, polystyrene, acrylic, polyurethane, and poly methyl acrylate. However, dense solutions of NaI or ZnBr_2 must be used to float polymers such as polyoxymethylene, polyethylene terephthalate, or polyester [20]. To remove unwanted organic material, sample digestion (post-treatment) with hydrogen peroxide (H_2O_2) or another chemical or enzymatic process is recommended [10]. Half of the studies (Table 1) used 15% H_2O_2 solution for sediment samples and 35% H_2O_2 solution for ice core samples. However, in this study a chemical digestion was unrequired due to the negligible amount of organic matter present in glacier samples.

Finally, the analysis of MP can be carried out by visual, spectrometric, spectroscopic identification, or by combining visual identification with one of the latter [11]. Visual identification with a microscope with μFTIR spectroscopic identification is the most likely approach to identify MP with a size between ~ 10 and $500\ \mu\text{m}$ [3,11]. Atmospheric MP used to be under $100\ \mu\text{m}$ and even below the μFTIR limit of detection (hereafter, LOD) ($11\ \mu\text{m}$) [2]. μFTIR spectroscopy with detection limits ranging from $11\ \mu\text{m}$ to $100\ \mu\text{m}$ has been used for the identification of MP polymers in surface snow samples and ice cores (Table 1). (Bergmann et al., 2019 [2]) and (Peeken et al., 2018 [5]) reported values with a LOD of $11\ \mu\text{m}$, so their results are up to 4 orders of magnitude greater than those with a LOD of $100\ \mu\text{m}$ [2,5]. We carried out the visual identification in a microscope with a LOD of $60\ \mu\text{m}$, which allows us to characterize the MP by color and shape having better results than those reported with larger LODs (Table 1). However, to carry out a more exhaustive polymer identification and to know other characteristics such as the polymer composition, spectroscopic identification is recommended.

4. Conclusion

In general, our technique has proven to be effective in all MP analysis stages. Sampling in mountain glaciers is complex due to accessibility. By using terrain-derived remote data for planning collection sites, we intend to optimize sampling by deciding how to do the sampling in advance, as the time of working at high altitudes is reduced and the relative costs of expedition are high. Our methodology was able to quantify and separate large amounts of MP, that vary in color and shape, in both surface snow and ice core samples. It does not require the use of abrasive substances and sieves, substantially reducing the preparation time and diminishing the risk of contamination by MP. By reducing the number of analytical steps, our method is less-manipulative, which is essential when working with an environmental matrix where a low level of MP contamination is expected.

When working in glaciers of high altitude mountains the volume of gear transported by each crew member is carefully planned, implying that additional sampling weight is limited. We expected low MP particles in a reduced volume of sample ($250\ \text{mL}$), but the abundance of MP of variable shapes and colors in contrast to blank samples was such that it confirms the effectiveness of our method, indicating that large volumes of samples are not strictly required for MP identification in glacial

environment matrices. Mountain glaciers are threatened ecosystems of high relevance in providing ecosystem services. The retreat of glaciers caused by climate change could release MP into watercourses and therefore, methodologies that enable to detect MP contamination over space and time are crucial.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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