Science of the Total Environment 870 (2023) 161942

Contents lists available at ScienceDirect

Science of the Total Environment

journal homepage: www.elsevier.com/locate/scitotenv

Experimental uncertainty assessment of meso- and microplastic concentrations in rivers based on net sampling

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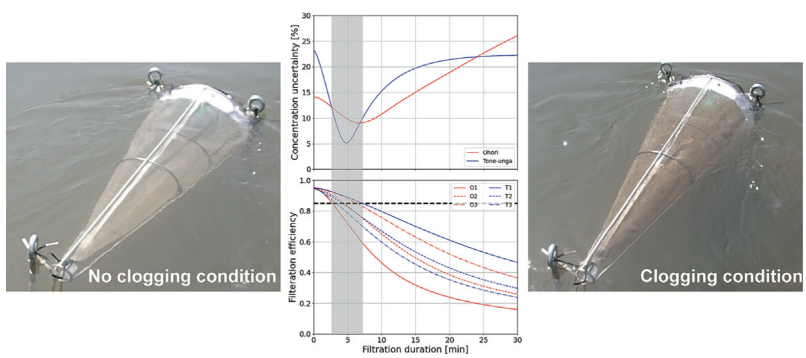
HIGHLIGHTS

- Uncertainty in plastic concentration measurements was experimentally examined.
- Thirty-two samplings with five filtration durations were conducted in urban rivers.
- The longer filtration duration yields more considerable concentration uncertainty.
- The sampling rate follows the Weibull reliability function (WRF).
- The plastic concentration and filtration efficiency can be expressed using WRF.

GRAPHICAL ABSTRACT



ARTICLE INFO

Editor: Dimitra A Lambropoulou

Keywords:
Uncertainty assessment
Plastic concentration
Filtration efficiency
Weibull reliability function
Net clogging
Urban rivers

ABSTRACT

Meso- and microplastics have been collected via net sampling in marine and freshwater environments, but the effect of net clogging on evaluations of their concentrations (mPC) remains uncertain. We experimentally investigated the mPC uncertainties resulting from net clogging in the Ohori and Tone-unga Rivers, typical urban rivers in Japan, throughout 16 samplings with five filtration durations in one day. The weighted mean concentration in the Ohori River was significantly lower than that in the Tone-unga River, allowing us to examine the effect of clogging in rivers with different contamination levels. The variances in both rivers consistently tended to increase with increasing filtration duration, which can be expressed by applying the integral form of the Weibull reliability function (WRF).

Furthermore, application of the WRF successfully revealed the optimal filtration durations in the Ohori and Tone-unga Rivers, which depended on the plastic abundance and sample volume. Since it could be difficult to obtain the plastic contamination level in advance, our suggestion is to predict the time sustained above 85 % filtration efficiency by applying a WRF-based model. In actuality, the sustained time in the Ohori (Tone-unga) River varied between 2.6 and 6.2 min (3.2 and 7.1 min) throughout the experiment, which permitted low mPC uncertainties of 12 % and 9.5 %, respectively. If notable uncertainty exists due to a low contamination level, a net with a high open area ratio should be used to increase the filtration duration. Hence, our results emphasize the importance of considering the open area ratio of nets used for sampling in studies. Our study provides insights into the occurrence of uncertainty due to net clogging to establish a standardized methodology for meso- and microplastic monitoring in aquatic environments via net sampling and consequently contributes to improving the sampling accuracy.

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http://dx.doi.org/10.1016/j.scitotenv.2023.161942
Received 3 November 2022; Received in revised form 28 December 2022; Accepted 27 January 2023
Available online 31 January 2023

1. Introduction

Tiny plastic particles called microplastics (<5 mm in size) have been collected from the water surfaces of seas, rivers, and lakes globally, mainly by towing plankton nets (Cózar et al., 2014; Kataoka et al., 2019; Law et al., 2010; Sighicelli et al., 2018). Quantifying plastic particles is an essential step for understanding their adverse impacts on marine and freshwater ecosystems and their environment-specific behaviors (Blettler et al., 2018; Mintenig et al., 2018). Net sampling is a common approach for collecting plastic particles from surface water more efficiently than using pump and bulk samplings and thus has often been used to evaluate plastic contamination in surface water (Watkins et al., 2021). However, net sampling-focused methodologies have not been sufficiently harmonized, resulting in comparison difficulties among different studies (Michida et al., 2019). Hence, several guidelines have been published recently to harmonize plastic particle sampling methodologies through net filtration (GESAMP, 2019; Michida et al., 2019).

According to these guidelines, various types and sizes of nets have been selected, mainly manta, neuston, and bongo nets (GESAMP, 2019; Michida et al., 2019). The net gauze mesh size of 200–333 μm has been most frequently selected, thus determining the detection size limit of collected plastics (GESAMP, 2019). Recently, several studies have examined how mesh size selection affects microplastic abundance estimations in marine environments (Lindeque et al., 2020; Tokai et al., 2021). As expected, these studies have demonstrated that relatively small-mesh net sampling results in large microplastic particle counts (Lindeque et al., 2020; Tokai et al., 2021; Weiss et al., 2021). However, small mesh nets should be used carefully because these nets can easily become clogged.

The effect of clogging on plastic particle quantifications during net sampling has still not been sufficiently debated under current guidelines, although its occurrence has been considered (Mendoza and Balcer, 2019; Michida et al., 2019). In particular, clogging could significantly affect microplastic sampling in rivers due to high turbidity and mixing (Bai et al., 2022; Michida et al., 2019). Thus, several researchers have adopted methods to avoid net clogging, for instance, setting short filtration (or tow) durations and using nets with relatively large mesh sizes (Bruge et al., 2020; Tanaka et al., 2022; Watkins et al., 2021). In contrast, short filtration durations may affect the plastic abundance representativeness because of the relatively low sample volume of filtered water (Prata et al., 2019). Here, a fundamental question arises: How should the filtration duration be determined? However, it is difficult to answer this question because the clogging occurrence conditions are unknown and because water clarity measurements do not aid in predicting the clogging rate (Fraser, 1968; Smith et al., 1968). As a consequence, to avoid clogging, continuous net sampling repetitions have been conducted (Bruge et al., 2020; Dris et al., 2018).

The filtration efficiency, i.e., the percentage of the velocity inside the net to that outside the net, should be considered to manage clogging conditions. In the 1960s, changes in the filtration efficiencies of plankton nets with different designs (types and mesh sizes) resulting from clogging during towing were investigated when sampling zooplankton from sea surface water (Smith et al., 1968). The accumulation of organisms on the net surfaces caused the water velocity through the net gauze aperture (i.e., mesh velocity) to decelerate due to a pressure drop (Tranter and Smith, 1968). Smith et al. (1968) considered a net to be clogged when the filtration efficiency fell below 85 %. Thus, 0.333-mm, 0.201-mm, and 0.101-mm mesh nets clogged 3, 5, and 35 times as rapidly as a 0.550-mm mesh net (Smith et al., 1968), indicating that the careless selection of nets with small mesh sizes may lead to unexpected clogging effects. Furthermore, the filtering-area-to-mouth-area ratio (i.e., the open area ratio) was found to be proportional to the sample volume under a constant mouth area at the logarithmic scale (Smith et al., 1968). Hence, sample volumes with 85 % or greater efficiencies can be increased sixfold by doubling the open area ratio (Smith et al., 1968). This suggests that the open area ratio is an important parameter dominating the filtration efficiency in sampling.

The present study aims to evaluate the effects of net clogging on plastic concentration measurements via meso- and microplastic collection through a total of 32 in situ samplings with five filtration durations in two small urban rivers (the Ohori and Tone-unga Rivers) in one day. In addition, to better understand the temporal variation in the clogging effect during each sampling, the sample volume decay per unit time (i.e., the sampling rate (SR)) was expressed by applying the Weibull reliability function (WRF), which can evaluate sample volume growth over time under net clogging via the integral form of the WRF SR model. This study provides suggestions for determining the filtration duration during net sampling in rivers as well as insights into the clogging mechanism through sampling.

2. Materials and methods

2.1. Sampling sites

Samples were collected from the water surfaces of the Ohori and Tone-unga Rivers in northern Tokyo on 26 March and 16 July 2020, respectively, under normal conditions (Fig. S1). The Ohori River (6.9 km long with a 31.0-km^2 basin area) connects to the mainstream Tone River, the largest river in Japan, via Lake Teganuma and the Tega River. The Tone-unga River (8.5 km long with a 25.4-km^2 basin area) is an artificial channel connecting the Edo and Tone Rivers. Urban areas account for 82 % and 50 % of the basins of the Ohori and Tone-unga Rivers, respectively, and the population densities are 6067 and 1333 persons/km^2, respectively (Nihei et al., 2020). The microplastic concentrations in the Ohori and Tone-unga Rivers are 12.9 and 12.7 particles/m^3, respectively (Nihei et al., 2020). These values are higher than the average values in Japanese rivers (4.3 particles/m^3; Nihei et al. (2020). Thus, both rivers are severely contaminated with microplastics, meaning that enough plastics can be collected to discuss the microplastic concentration differences among different filtration durations.

The Ohori River sampling site is located 4.1 km upstream from the confluence with Lake Teganuma (O in Fig. S1), and that of the Tone-unga River is located 2.5 km upstream from the confluence with the Edo River (T in Fig. S1). These dimensions are shown in Fig. S1. Water levels were measured with reference to Tokyo Pail by the Ministry of Land, Infrastructure, Transport and Tourism (http://www1.river.go.jp/) (Fig. S2).

2.2. Sampling experiment

Samples were collected using a conical plankton nylon net (No. 5512-C; RIGO Co. Ltd., Japan); the net mouth diameter, mesh width (d), and net length were 30 cm, 335 μm, and 75 cm, respectively (Fig. S1). The gauze porosity (β) was 0.45, calculated from $d^2/(\phi + d)^2$, where ϕ is the meshwork strand diameter (165 μm) (Tranter and Smith, 1968). The open area ratio was 2.41, calculated from $s\beta/a$, where s and a are the gauze surface area and the projected net mouth area (Tranter and Smith, 1968), which were 0.379 m^2 and 0.0707 m^2 ($=0.30^2 \times \pi/4$), respectively. The net was of the same material as that used in our previous works (Kataoka et al., 2019; Nihei et al., 2020). A flow meter with a speed range of approximately 0.06–1 m/s (2030R6, General Oceanics Inc., USA) was installed at the net mouth to grasp the sample volume and SR. To maintain net straining regardless of the flow state of the river, a customized stainless frame was equipped on the outside of the net. In addition, to avoid any unexpected trouble, such as the blade of the flow meter touching the net, a steel frame was also equipped on its interior. To fix the vertical and horizontal locations of the net, the net was set on the surface by three 1.2-m steel stakes and thus not towed in this study (Fig. S1). Finally, the net mouth was covered by a customized 100 % cotton cover until sampling commenced.

As the filtration duration has often been selected to be 5 min in previous works (Kataoka et al., 2019; Nihei et al., 2020), five cases consisting of durations shorter and longer than 5 min were determined (i.e., 3, 5, 7, 10, and ≥20 min). In the longest case (≥20 min), sampling was continued until the flowmeter stopped due to the clogging effect. These five cases were packaged and then repeated in triplicate. The order of five cases in three packages was unified to arrange the temporal interval between samplings with each filtration case as much as possible, which could make it easy to interpret the temporal effect of river condition change during the experiment. Moreover, each package started from the sampling of the 5-min filtration case, and the 5-min case sampling was conducted after the third package (Table S1). Thus, 32 case samplings were carried out in total in both rivers. The flowmeter stopped rotating at approximately 20 min in most cases but kept rotating after 20 min in the first package of the Tone-unga River (T1); thus, the ≥20-min T1 case was sampled for 30 min. After each sampling, the net mouth was covered by a customized cover, and the net was brought back to the laboratory.

The sample volume was estimated by multiplying the net mouth area (a = 0.0707 m^2) by a filtration distance, calculated as follows: (rotation counts of flowmeter) × (57,560: rotor constant for 2030R6)/999,999). SR was estimated only in the ≥20-min case of O2 and T3 by measuring seconds until five flowmeter blade rotation counts occurred at one-minute intervals to understand the temporal decay. Because turbidity is expected to be related to net clogging, turbidity was measured at intervals between two packages (Fig. 1a and b) using a Compact-CTD instrument (ASTD687, JFE Advantech Co. Ltd., Japan). The turbidity data in the Formazin turbidity unit (FTU) were recorded for 1 min at 0.1-s intervals on the water surface and then averaged over 1 min (Fig. 1a and b). In addition, to confirm the temporal flow state change, the surface current velocities (depth < 0.3 m) were measured at the start and end of each experiment with an electromagnetic current meter (LP40, Kenek Co., Ltd., Japan) or a float method in which the time until a floating object passed a 1-m distance was measured (Table S2).

2.3. Laboratory analysis for identifying meso- and microplastics

After the in situ sampling experiment, all matter collected by the net was temporarily transferred to 500-ml stainless-steel bottles in our laboratory by rinsing the net using tap water filtered by a 100-μm mesh nylon net. Then, the samples were filtered by a magnetic filter funnel (500-ml volume, 47-mm filter size) attached to a 100-μm nylon net as a filter. The filter samples were placed on a glass Petri dish with a glass lid and dried at 60 °C for at least 24 h. Thereafter, the dried samples were transferred to a 100-ml glass beaker, and 50 ml of 30 % hydrogen peroxide (H_2O_2) solution was poured into the beaker to degrade natural organic matter at room temperature for at least one week. After one week, the residual matter was collected by the funnel again and then dried at 60 °C for at least 24 h.

Particles that could possibly be plastic were picked up with a stainless-steel tweezer from filtered samples by visual observation (Hidalgo-Ruz et al., 2012). To remove bias in the analysis, this process was conducted by two different analysts. Then, each particle was observed by a

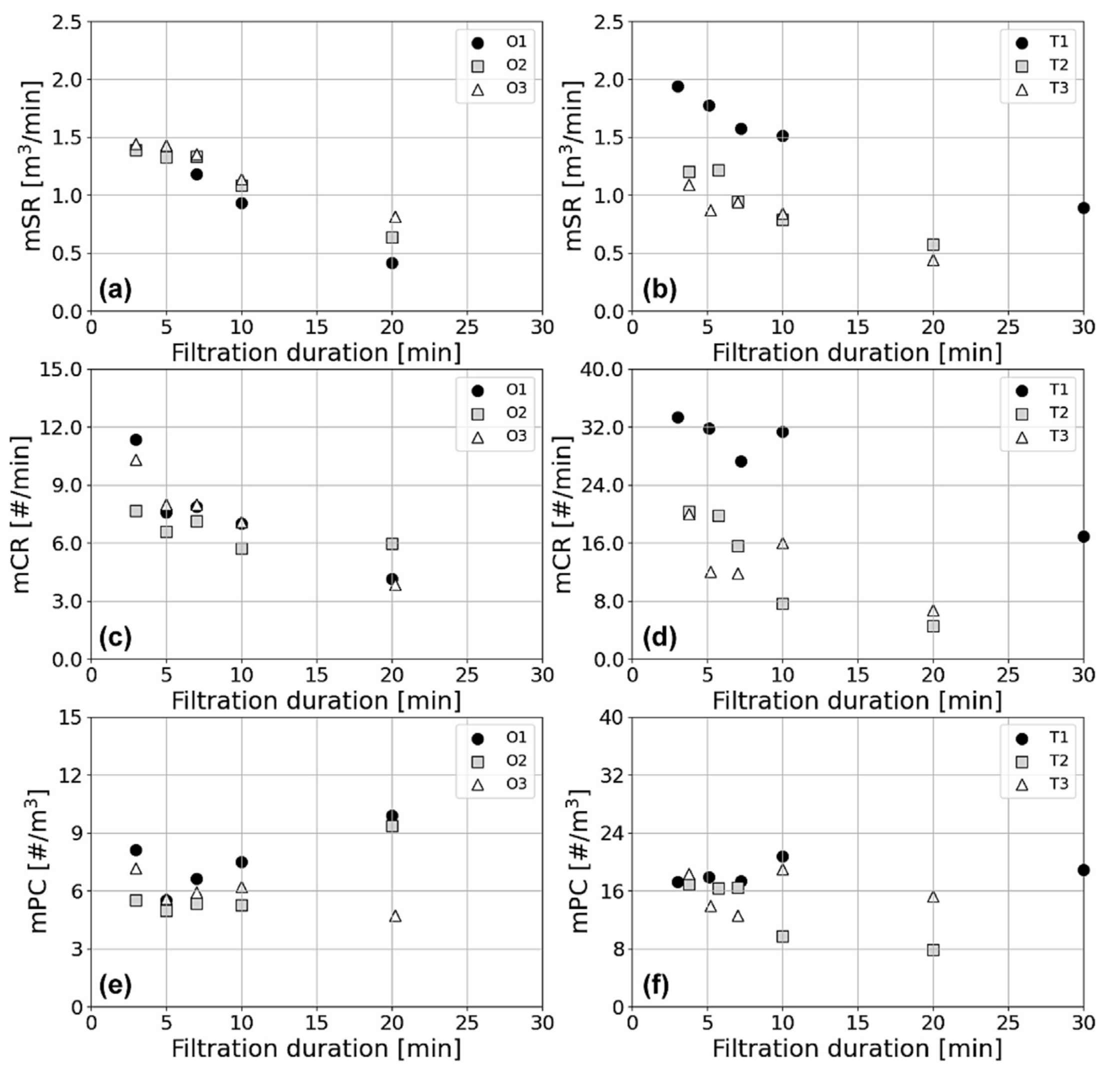

Fig. 1. Decrease in mSR ((a) and (b)), mCR ((c) and (d)), and mPC ((e) and (f)) depending on filtration duration. The left- and right-side panels show the Ohori and Tone-unga Rivers, respectively. The legend is shown in the upper-right box in each panel, and the three symbols indicate the different packages.

stereoscopic microscope (SZX7, Olympus Co. Ltd., Japan) installed with a USB camera (HDCE-20C; AS ONE Co. Ltd., Japan), its shape was categorized, and its size was measured. Particles were categorized into five shape types (pellet, sphere, fragment, fiber and sheet). The maximum particle length was measured using ImageJ image analysis software (https://imagej.nih.gov/ij/) to clarify the size distribution of the plastics and differentiate meso- and microplastics.

Finally, the material of each particle was identified by Fourier transform infrared spectrophotometry (FTIR: IRAffinity-1S, Shimadzu Co. Ltd., Japan) using installed attenuated total reflection (ATR) equipment (Quest, Specac Ltd., Japan). The IR absorption spectrum was measured by scanning from 500 to 4600 cm^{-1} at a 4-cm^{-1} resolution; then, the polymer type was identified by comparison with the reference IR spectra stored in three spectral libraries (ATR-Polymer2, Irs-Polymer2, T-Polymer2) provided by Shimadzu Co. Ltd. The hit quality index (HQI) ranged from 0 to 1000 ‰. To avoid polymer type misidentifications, we set 700 ‰ HQI as the threshold value.

2.4. Quality assurance and control of in situ net sampling and laboratory analysis

To prevent contamination with unexpected plastics, several measures were established during the in situ sampling experiments and laboratory analyses. At the sampling site, the net mouth was always covered with a 100 % cotton cloth except during sampling. The tap water used during laboratory analyses was filtered with a 100-μm nylon net. Since the nylon net was used during sampling and laboratory analysis, 24 nylon particles identified by the FTIR analysis were ignored. In laboratory analysis, we wore 100 % cotton robes worn and avoided using plastic utensils (e.g., beakers, Petri dishes, and tweezers). In addition, a blank test was conducted to grasp the airborne contamination by collecting particles deposited from the air in a room with disposal Petri dishes (ϕ 91 mm) on the tables during each sample analysis. After analyzing each sample, FTIR was used to identify all deposited particles, and the same spectral libraries and threshold HQI were applied to analyze the relevant blank test (see 2.3). As a result, eight unexpected plastic particles (seven fibers and one fragment) were found in 5 of 32 cases (Table S1). After these unexpected particles were cross-checked with the color, shape and polymer type of all plastic particles in the same case, no doubtful plastic particles were found, indicating that no unexpected plastic contamination occurred during the laboratory analyses.

2.5. Modeling of the sampling rate decay due to net clogging via the Weibull distribution

The deterioration in the filtration efficiency due to net clogging can be described as the survival time from net sampling initiation until complete net clogging (failure of net sampling). In this study, the temporal decay in SR was expressed via the following equation with three parameters (q_0, α and τ_q) based on the Weibull reliability function (WRF), denoted as $R(t)$ in Text S1.

$$q(t) = q_0\ exp\left[-\left(\frac{t}{\tau_q}\right)^{\alpha}\right] = q_0 R(t) \quad (1)$$

where α is the shape parameter determining the probability distribution form, τ_q is a scale parameter with time dimensions, and q_0 is the initial flow rate through the net, which can also be expressed as $q_0 = u_0 a$, where u_0 is the initial cross-sectional mean velocity at the net mouth and a is the mouth area. Since the net was fixed on the river surface, u_0 corresponds to the surface flow velocity considering the initial filtration efficiency determined via the empirical equation suggested by Tranter (1967) (Text S2). Parameters α and τ_q can be determined considering SR measured by counting the number of rotations of the flowmeter in O2 and T3 through regression analyses applied to the nonlinear least squares method.

2.6. Statistical analyses

Three metrics were defined to examine the net clogging effect: the mean sampling rate (mSR; in units of m^3/min) $\overline{q}_{i,j}$, mean plastic capture rate (mCR; #/min) $\overline{r}_{i,j}$, and mean plastic concentration (mPC; #/m^3) $\overline{c}_{i,j}$ observed in the i^{th} case ($i = 1, 2, \cdots, 5$) in the j^{th} package $j = 1, 2, 3$:

$$\overline{q}_{i,j} = \frac{V_{i,j}}{t_{i,j}} \quad (2)$$

$$\overline{r}_{i,j} = \frac{A_{i,j}}{t_{i,j}} \quad (3)$$

$$\overline{c}_{i,j} = \frac{A_{i,j}}{V_{i,j}} \quad (4)$$

where $A_{i,\,j}$, $V_{i,\,j}$, and $t_{i,\,j}$ are the number of mesoplastics and microplastics collected with the net (i.e., plastic abundance), sample volume, and filtration duration, respectively. mSR and mCR denote the averages of the flow rate and plastic flux passed through the net during sampling, respectively. When calculating the mean and variance in these three values, we considered their relative significance (Tanaka et al., 2022). We expected that the more river water was sampled (or the longer the filtration duration was), the greater the net clogging effect would be. Therefore, the weighted means of these values were calculated using $V_{i,\,j}$ or $t_{i,\,j}$ as follows:

$$m_q = \Sigma_{i,j}\overline{q}_{i,j}w\prime_{i,j} \quad (5)$$

$$m_r = \Sigma_{i,j}\overline{r}_{i,j}w\prime_{i,j} \quad (6)$$

$$m_c = \Sigma_{i,j}\overline{c}_{i,j}w_{i,j} \quad (7)$$

where $w\prime_{i,\,j}$ and $w_{i,\,j}$ are the weight of each value computed by $t_{i,j}/\Sigma_{i,j}t_{i,j}$ and $V_{i,j}/\Sigma_{i,j}V_{i,j}$, respectively. The weighted variance was then calculated:

$$\sigma_q^2 = \frac{N}{N-1}\Sigma_{i,j}\left(\overline{q}_{i,j} - m_q\right)^2 w\prime_{i,j} \quad (8)$$

$$\sigma_r^2 = \frac{N}{N-1}\Sigma_{i,j}\left(\overline{r}_{i,j} - m_r\right)^2 w\prime_{i,j} \quad (9)$$

$$\sigma_c^2 = \frac{N}{N-1}\Sigma_{i,j}\left(\overline{c}_{i,j} - m_c\right)^2 w_{i,j} \quad (10)$$

where N means the number of replicas, and $N/(N-1)$ denotes the transformation to unbiased variance. To examine the value differences among different filtration cases (packages), the weighted means and variance in the i^{th} case (j^{th} package) were computed by averaging the values over three packages on each case (five filtration cases on each package), as shown in Eqs. (5)–(7) (e.g., $m_{ci} = \Sigma_j\overline{c}_{i,j}w_{i,j}$, where $w_{i,j} = V_{i,j}/\Sigma_j V_{i,j}$). The uncertainty of each value can be empirically evaluated/predicted with the precision (p), defined using the weighted mean and variance:

$$p = \frac{\sigma}{\sqrt{N}m} \quad (11)$$

where m and σ are provided by Eqs. (5)–(7) and the square root of Eqs. (8)–(10), respectively. p is the standard error ($SE = \sigma/\sqrt{N}$) normalized by the mean; this term can be used to evaluate the mean error range. p suggests that the higher the number of samples is, the lower the confidence interval is; thus, many studies commonly collect replicas through multiple net samplings to reduce the estimate uncertainty (e.g., Bruge et al., 2020).

As the Shapiro–Wilk test rejected the null hypothesis that the three metrics in each sampling package and filtration case were normally distributed at the 95 % confidence level, the differences in the three metrics among the three packages and five filtration cases were compared by applying generalized linear models (GLMs). For mPC and mCR, the Poisson distribution with a log link function was used because the response variable was determined to be the number of particles, that is, count data. The log of sample volume for mPC and filtration duration for mCR were included in the log link function as an offset. For mSR, the gamma distribution with a log link function to which the log of filtration duration was added as an offset was used. The results from the GLM fits were presented as analysis of deviance tables showing the difference among the sampling packages and filtration cases. The hypothesis tests in the tables were performed using type I model comparisons. When a significant difference was found, Tukey's HSD test was performed as post hoc statistical analysis for an all-pairwise multiple comparison procedure. For the discussion of the clogging effect, Pearson product moment correlation analysis was performed to statistically compare the three metrics estimated through nonlinear regression analysis with those observed. Additionally, to determine the optimum filtration duration in the discussion, a linear regression analysis was applied. All statistical analyses were performed in R (version 4.1.2, R Development Core Team) with packages “multcomp” and Python (version 3.8.8) with the library “scipy” version 1.6.2. The statistical significance was reported at $p = 0.05$.

3. Results

3.1. Temporal variation in the mean sampling rate

Through the in situ experiment, 136.8-m^3 and 143.1-m^3 sample volumes were filtered from the Ohori and Tone-unga Rivers for total durations of 140.25 and 152.82 min, respectively (Table S3). The weighted mSR means ($m_q \pm \sigma_q$) in both rivers were also similar (Table S3). In both rivers, mSR declines were seen in the longer filtration cases (e.g., the ≥20-min case) in all packages (Fig. 1c and d), resulting from the filtration efficiency decay due to the occurrence of net clogging. Furthermore, the temporal mSR variation differed between the two rivers. In the Ohori River, the mSRs in the 3-min and 5-min cases hardly changed among packages, while those in the other cases significantly increased (Fig. 1a), indicating that the filtration efficiency improved over packages. In contrast, the mSRs in all cases decreased over packages in the Tone-unga River (Fig. 1b), indicating that the filtration efficiency in the even shorter filtration case (e.g., 3-min case) significantly declined.

3.2. Temporal variations in mean plastic capture rate and concentration

In the water samples from the Ohori and Tone-unga Rivers, 874 and 2334 plastic particles were found (Table S3), and 89 % and 96 % of the collected plastics were microplastics (i.e., < 5 mm), respectively; the rest were meso-sized (Fig. S3). The fragment plastic particle type was predominant in both rivers (65 %: Ohori River, 86 %: Tone-unga River), and the rest were mostly categorized as the fiber type (Fig. S3).

The mCR in the Tone-unga River varied more than that in the Ohori River, indicating that the precision of the Tone-unga River (14 %) was greater than that of the Ohori River (7 %) (Table S3). Indeed, compared to the mCR in the Ohori River, that in the Tone-unga River largely varied over time (Fig. 1c and d). In particular, the mCR in the longer filtration case (e.g., ≥20-min case) was significantly lower than that in the shorter cases (e.g., 3-min and 5-min cases).

The mPCs of the Ohori River and the Tone-unga River ranged from 5.0 to 10 #/m^3 and 7.9–21 #/m^3, respectively (Fig. 1e and f; Table S1). On average, the $m_c \pm \sigma_c$ of the Tone-unga River was approximately 2.5 times greater than that of the Ohori River, indicating that the Tone-unga River was highly contaminated by plastics (Table S3). Nevertheless, it is interesting that the precision of the Ohori River was similar to that of the Tone-unga River (Table S3). This indicates that the variance in the mean concentration measured by repeated sampling was the same in the two rivers.

3.3. Weighted mean of and variance in the three metrics in each package and case

The weighted means of the three metrics (mSR, mCR, and mPC) in the three packages (m_{qj}, m_{rj} and m_{cj}) exhibit temporal variations throughout the experiment, and those in the five cases (m_{qi}, m_{ri} and m_{ci}) exhibit differences among filtration durations (Fig. 2). The m_{qj} of the Tone-unga River significantly decreased over time, and the pairwise comparison found a significant difference from T1 (Fig. 2a), indicating that the flow condition temporally varied in T2 resulting from the increased water level (Fig. S2). The temporal flow change also yields a statistically significant decrease in m_{rj} (Fig. 2b), diluting the plastic concentration in the water surface layer. In accordance with the difference in m_{qj} and m_{rj}, there was a significant difference in all pairwise comparisons of m_{cj} (Fig. 2c). Meanwhile, a significant m_{ri} difference was found, but m_{ci} was not significantly different among the five cases (Fig. 2e and f).

In contrast, for the Ohori River, there were no significant differences in m_{qj} and m_{rj}, indicating that their changes were predominant with the difference in filtration duration rather than in temporal variation (Fig. 2a-b). In fact, the pairwise comparisons found significant differences in m_{qi} and m_{ri} (Fig. 2d and e). In particular, it is interesting that mSR and mCR in the longer filtration durations (10 min or ≥ 20 min) were significantly different from those in the shorter filtration durations (3 min or 5 min) (Fig. 2d and e), indicating considerable decreases due to clogging. Consequently, a significant m_{ci} difference was found among the five cases, in particular, the difference between the 5-min and ≥ 20-min cases (Fig. 2f).

Interestingly, in both rivers, mPC tended to vary as the filtration duration elongated, and its variance was greatest in the ≥20-min case (Fig. 3). The minimum precisions in the Ohori and Tone-unga Rivers occurred in the 5-min and 3-min cases, respectively, in which the net clogging effect could be relatively low. The 3-min-case precision in the Ohori River was greater than that in the Tone-unga River, indicating that variance was induced by the low representativeness (plastic abundance) and that unexpected mPC variance occurs when the sample volume is not sufficient to evaluate mPC, as in the Ohori River 3-min case (Fig. 3a).

Such mPC uncertainty can be demonstrated from the different mSR and mCR precisions among the five cases. In the Ohori River, the mSR precision increased with the filtration duration, indicating that net clogging contributes to mSR variation (Fig. 3a). Moreover, mCR decreased until the 7-min filtration duration but increased in the longer filtration cases (i.e., 10-min and ≥ 20-min cases). In the 3-min case, the mCR precision was greater than the mSR precision and was similar to that of mPC. In contrast, the mSR precision was greater than that of mCR in the ≥20-min case, and thus, the mSR variance dominated the mPC variance rather than mCR. On the other hand, in the Tone-unga River, no mSR precision differences were found among the five cases (Fig. 3b). The large mSR variance regardless of the filtration duration could be associated with the temporal changes in flow conditions in T2 (Fig. S2). As a consequence, the mPC precision was dependent on the mCR variance and monotonically increased as the filtration duration increased (Fig. 3b). Thus, it could be helpful to consider the mSR and mCR precision levels when assessing the factors determining the mPC variance.

3.4. Modeling of the sampling rate and volume using the Weibull reliability function

While sampling water in O2 and T3, SR significantly decreased due to net clogging. The temporal SR decay in O2 and T3 (Fig. S4) can be approximated very well by the WRF through nonlinear regression analysis (Table 1). The q_0 values in the two packages were 1.56 m^3/min and 1.15 m^3/min, corresponding to 0.0260 m^3/s and 0.0192 m^3/s, respectively. Hence, the u_0 values in O2 and T3 were 0.369 m/s and 0.271 m/s, respectively. These velocities correspond to the surface velocities of the rivers because the initial filtration efficiency was approximately 0.95 (Text S2).

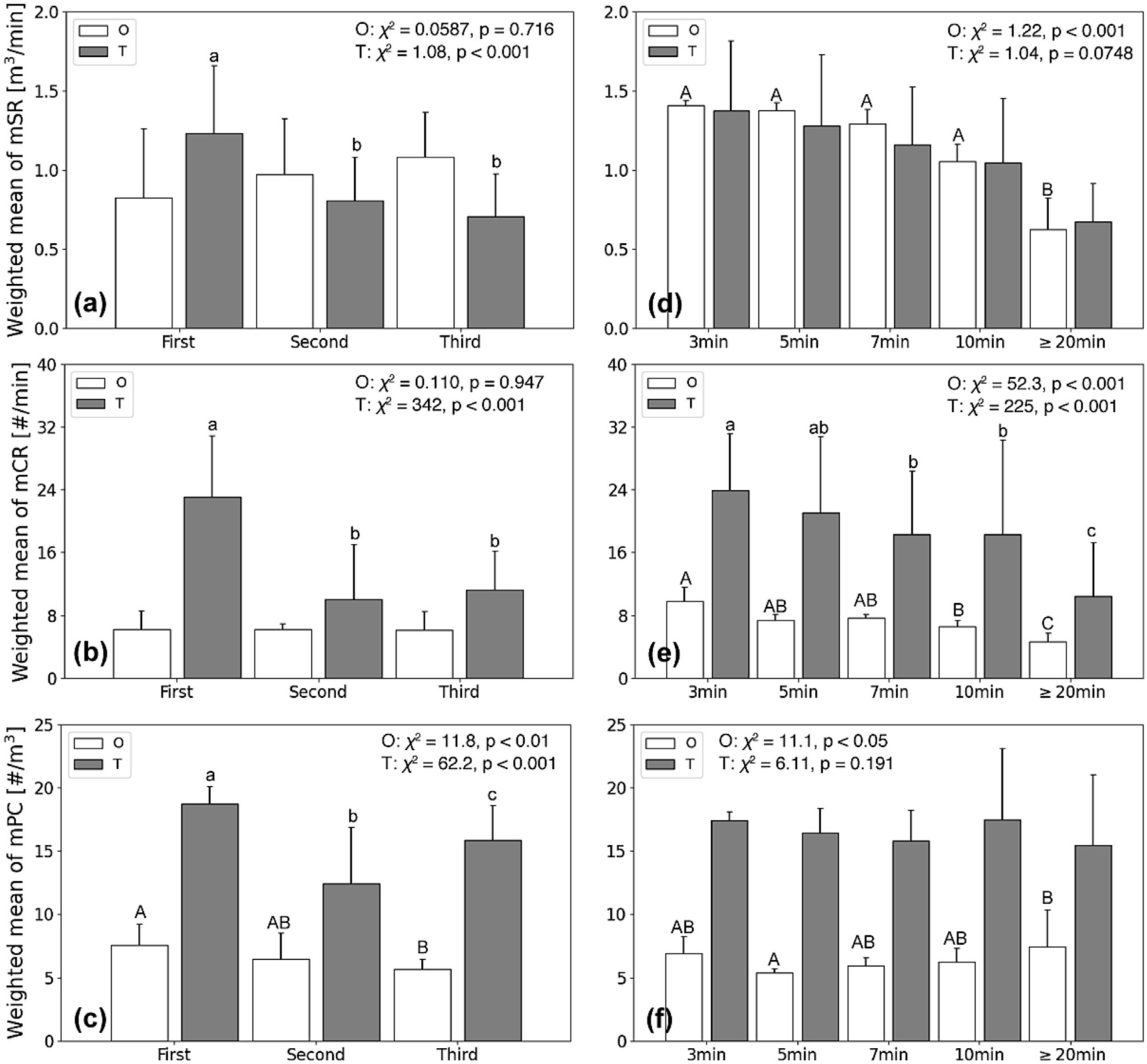

Fig. 2. Weighted mean mSR, mCR, and mPC concentrations under the five filtration durations in each package ((a), (b), and (c), respectively) and over the three packages under each filtration duration ((d), (e), and (f), respectively). In each panel, the error bar indicates the standard deviation (1σ). When statistical significance was determined via one-way ANOVA, as shown in each panel, Tukey's test was performed as a post hoc pairwise comparison. The legend is shown in the upper-left box in each panel, and O and T denote the Ohori and Tone-unga Rivers, respectively. The same letter (uppercase for O; lowercase for T) indicates that there was no significant difference at $p = 0.05$.

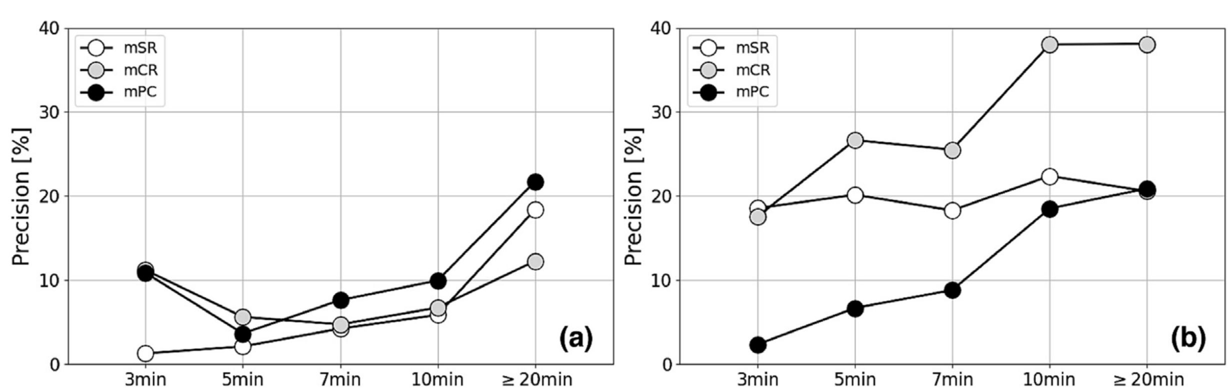

Fig. 3. Dependence of the precision for the observed mSR, mCR and mPC in the Ohori (a) and Tone-unga (b) Rivers under the five filtration durations.

Table 1
Three WRF parameters estimated from the observed sampling rate, sample volume and plastic abundance.

	O1	O2	O3	T1	T2	T3
q_0 [m^3/min]	1.79	1.58 (1.56)	1.49	1.82	1.24	1.22 (1.15)
u_0 [m/s]	0.42	0.37 (0.37)	0.35	0.43	0.29	0.29 (0.27)
τ_q [min]	5.64	9.18 (9.18)	13.03	17.16	10.46	8.37 (8.26)
α		1.66			1.45	
r^2	0.926	0.991 (0.984)	0.995	0.998	0.922	0.944 (0.982)
r_0 [#/min]	9.74	6.54	10.17	34.50	40.53	19.32
τ_r [min]	9.67	43.3	8.76	17.22	2.61	8.47
α		1.66			1.45	
r^2	0.961	0.986	0.979	0.988	0.120	0.665

Note: The values in the parenthesis denote the parameters estimated from the temporal SR decay observed in O2 and T3.

Hence, the surface velocity in the ≥20-min of O2 was faster than that of T3. Moreover, the T3 scale parameter τ_q was slightly shorter than that of O2, indicating that the river water of the latter case could be more efficiently filtered than that of the former. Consistently, both shape parameters ranged between 1 and 2, meaning that the hazard function ($\lambda(t)$in Eq. (S3)) increased over time (positive aging process).

To examine the applicability of WRF for SR (Eq. (1)), the sample volumes in all O2 and T3 cases were estimated by integrating over the filtration duration as follows:

$$V(t) = \int_0^t q_0 \, exp\left[-\left(\frac{\tau}{\tau_q}\right)^{\alpha}\right] d\tau \tag{12}$$

The results were compared with the observed values (Fig. S4). This model can reproduce the sample volumes in both packages ($r = 0.987$, $p < 0.01$), suggesting that the single-package sample volume can be predicted by the integral WRF form with the same regression parameters.

4. Discussion

Relatively long filtration durations yield unexpected mSR and/or mCR variance due to net clogging, resulting in high mPC uncertainty. Additionally, a river with a low contamination level would have a higher mPC variance in the short filtration cases; as a result, the Ohori River mPC uncertainty was relatively high. Furthermore, the temporal growth of the sample volume in each package can be approximated by WRF. Here, we discuss the sampling methodology under net clogging conditions by representing the temporal mPC variation using WRF. WRF can fully determine the most appropriate filtration duration in the two rivers and finally answer the question "How long should we sample water to obtain reliable data in net sampling?"

4.1. Evaluation of temporal flow and clogging condition changes

The applicability of WRF (Fig. S4) can allow us to estimate the three regression parameters by approximating the sample volumes in the five cases in each package by Eq. (12) through nonlinear regression analysis. First, the three parameters were estimated by Eq. (12) using the sample volume only as a trial (Table S4). Unexpectedly, T2 was unable to find the optimal parameter. Furthermore, α in T1 was smaller than one, indicating a negative aging process in which $\lambda(t)$ in Eq. (S3) was reduced from positive infinity at $t = 0$ and then monotonically decreased (Rinne, 2008). However, $\lambda(t)$ for clogging should be considered a positive aging process ($\alpha > 1$) in which the greater age is reached, the higher the probability of clogging is. In addition, $\lambda(t)$ could eventually converge considering that the net was fully clogged. If so, it is likely to be $\alpha < 2$ because the derivative of $\lambda(t)$ becomes zero when $t \rightarrow \infty$. Based on this suspicion, compared to the other parameters, α is likely to vary between 1 and 2 and thus could be insensitive to temporal flow and clogging condition changes.

For stability during parameter estimation, we assumed that α of each river was constant among packages; then, we imposed temporal flow and clogging condition variations on q_0 and τ_q, respectively. Under this assumption, the two parameters were successfully estimated from the observed sample volume in each package through nonlinear regression analysis (Table 1). Compared to the estimates from the temporal SR decay, q_0 and τ_q in both O2 and T3 were slightly calibrated by the sample volume data in the other cases (Table 1). The temporal growth of the sample volume can be approximated very well by Eq. (12) (Table 1; Fig. S5).

Both rivers became gentler over time through the experiment, particularly in the Tone-unga River, supported by the measured current velocity (Table S2). Moreover, the τ_q of the Ohori (Tone-unga) River increased (decreased) over packages, indicating that the filtration efficiency increased (decreased). Consequently, the mSR of the Ohori (Tone-unga) River increased (decreased) over time (Fig. 1a and b). From the temporal τ_q changes in the Ohori and Tone-unga Rivers, we suspect that the turbidity decreased and increased over time, respectively. However, the turbidity measured in the Ohori River hardly varied (Fig. S2), and in the Tone-unga River, it decreased (Fig. S2) regardless of the temporal variation in τ_q. As mentioned by Smith et al. (1968), this contradiction emphasizes that the water clarity measurement is not useful in predicting the clogging rate.

4.2. Expression of temporal plastic abundance growth using the Weibull reliability function

If plastic particles can be assumed to be randomly distributed in the water column (homogeneity) and to be constantly transported with the flow during each package (stationarity), the temporal growth of the plastic abundance can be expressed using the integral form of WRF as follows:

$$A(t) = \int_0^t r_0 \, exp\left[-\left(\frac{\tau}{\tau_r}\right)^{\alpha}\right] d\tau \tag{13}$$

where $A(t)$, r_0 and τ_r are the temporal growth of plastic abundance, the initial plastic capture rate, and the time until the capture rate decreases to r_0 exp (-1), respectively. Here, we assumed that α is consistent with that in Eq. (12) because it would be determined by the net properties (open area ratio and gauze porosity) and clogging condition changes. According to the manner of approximating the sample volume, the two parameters (i.e., r_0 and τ_r) were estimated from the plastic abundance data in each package. The temporal growth of plastic abundance, except in T2 and T3, can be expressed very well by Eq. (13) (Table 1; Fig. S5). Unfortunately, it was difficult to express the temporal growth in T2 and T3 due to the variation in plastics suspended in the water column induced by flow condition changes in T2 (Fig. 1b). In particular, mCR and mPC rapidly decreased in the 10-min and ≥ 20-min cases of T2 because the plastics on the surface were mixed due to turbulence enhanced by the flow change (Fig. 1b) and were then diluted. Consequently, turbulent mixing could impose the larger mPC variations observed in T2 and T3 (Fig. 1f).

Regardless of the different sampling conditions in each package, it is interesting that the temporal plastic abundance growth can be expressed by certain parameters. A number of studies have discussed the large mPC variations arising due to an unsteady river environment state induced by hydrodynamics, such as wind, currents and turbulence (Cook et al., 2020; Kooi et al., 2018; Mintenig et al., 2020; Uzun et al., 2022). Indeed, the behaviors of plastics are sensitive as plastic concentrations fluctuate rapidly and mix due to the slight flow change that occurs in T2. Nonetheless, our results support that the steady plastic transport state that met the above two assumptions (i.e., homogeneity and stationarity in plastic transport) existed during the few hours in which single-package sampling was conducted.

Furthermore, these results emphasize that replicated sampling should be completed within as short of a duration as possible to reduce the occurrence of mPC uncertainty induced by clogging. In other words, we should not consider a couple of samples collected under the condition that the mPC variance is too large among the replicated samples due to the clogging effect, even if the total sampling duration was within a few hours. To validate replicated samples, our suggestion is to examine the decay in filtration efficiency during each sampling, as discussed in 4.4. Additionally, our results showed that the variance was large due to the change in flow conditions, such as T2, indicating that the validity of the replicated sample would depend on the change in flow conditions, such as flooding. In this case, we would make it difficult to collect replicated samples under flow changes.

4.3. Dependence of plastic concentration on filtration duration

By applying the integral form of WRF for the sampling volume (Eq. (12)) and plastic abundance (Eq. (13)), the mPC on each package can be expressed using the following equation (Fig. 4a and b):

$$\bar{c}(t) = \frac{A(t)}{V(t)} = \frac{\int_0^t r_0 \, exp\left[-\left(\frac{\tau}{\tau_r}\right)^{\alpha}\right] d\tau}{\int_0^t q_0 \, exp\left[-\left(\frac{\tau}{\tau_q}\right)^{\alpha}\right] d\tau} = c_0 \frac{\int_0^t \, exp\left[-\left(\frac{\tau}{\tau_r}\right)^{\alpha}\right] d\tau}{\int_0^t \, exp\left[-\left(\frac{\tau}{\tau_q}\right)^{\alpha}\right] d\tau} \quad (14)$$

where $c_0(=r_0/q_0)$ is the concentration free from the effect of clogging. $\bar{c}(t)$ in both rivers can represent the observed mPC very well ($r = 0.960$, $p < 0.01$). The mPC in O1 and O2 increased as the filtration duration increased, while that in O3 decreased (Fig. 4a). In the Tone-unga packages, the mPCs in T1 and T3 were constant despite the different filtration durations, while that in T2 decreased. In particular, filtration durations longer than 10 min yielded enlarged mPC variations in both rivers. According to our recent work (Tanaka et al., 2022), the weighted mPC mean (m) was calculated by Eq. (10) using the data from all the samplings ($N = 10$) through filtration durations shorter than 10 min, that is, 3, 5, and 7 min ($\times$ on the left vertical axis of Fig. 4a and b). If the mPC was relatively varied between 10 % and 30 %, mPC had the ranges with gray in Fig. 4a and b by applying the coefficient of variation (CV: σ/m). The mPC observed by the shorter-filtered sampling was within the 10 %-gray range against the weighted mean, while those observed by the longer-filtered sampling exceeded the 10 %-gray range. Conversely, shorter-filtered sampling in a river with a relatively low contamination level would also yield mPC uncertainty.

This mPC uncertainty was supported by the continuous change in mPC precision computed by substituting the mean (m_c of Eq. (7)) and standard deviation (σ_c of Eq. (10)) at time t calculated by applying $\bar{c}(t)$ and $V(t)$ of three packages ($N = 3$) into Eq. (11). In fact, the mPC precision increased under both short and long filtration durations (Fig. 4c and d, respectively). Consequently, the mPC precision (9.4 and 5.1) was minimized at 6.6 and 4.8 min in the Ohori and Tone-unga Rivers, respectively, suggesting the most appropriate filtration durations under the given river conditions.

Recently, Tanaka et al. (2022) proposed the following variance–mean relationship:

$$\sigma^2 = \alpha_1 m^{\alpha_2} \quad (15)$$

where α_i ($i = 1, 2$) are empirical parameters corresponding to the intercept and slope on a logarithmic scale, respectively. Substituting Eq. (15) into Eq. (11), the projected precision is derived:

$$p = \sqrt{\frac{\alpha_1}{N}} m^{\alpha_2/2-1} \quad (16)$$

Assuming $\alpha_2 \approx 1$ (Tanaka et al., 2022), the precision is proportional to $m^{-1/2}$ and hence decreases as mPC increases (Fig. 5 in Tanaka et al. (2022)). Thus, since plastics are abundant in the water column of the Tone-unga River, the observed mPC had a relatively low uncertainty even if the filtration duration was short (Fig. 3b). In addition, the minimum mCR precision in the Ohori River (12 min) was greater than that in the Tone-unga River (2.3 min) (broken line in Fig. 4c and d). This implies that compared to the Tone-unga River, longer-filtered sampling is needed to reduce the mCR uncertainty in the Ohori River. In contrast, this would cause mSR uncertainty due to net clogging (dashed line in Fig. 4c and d), suggesting that the filtration duration required to lower the mPC

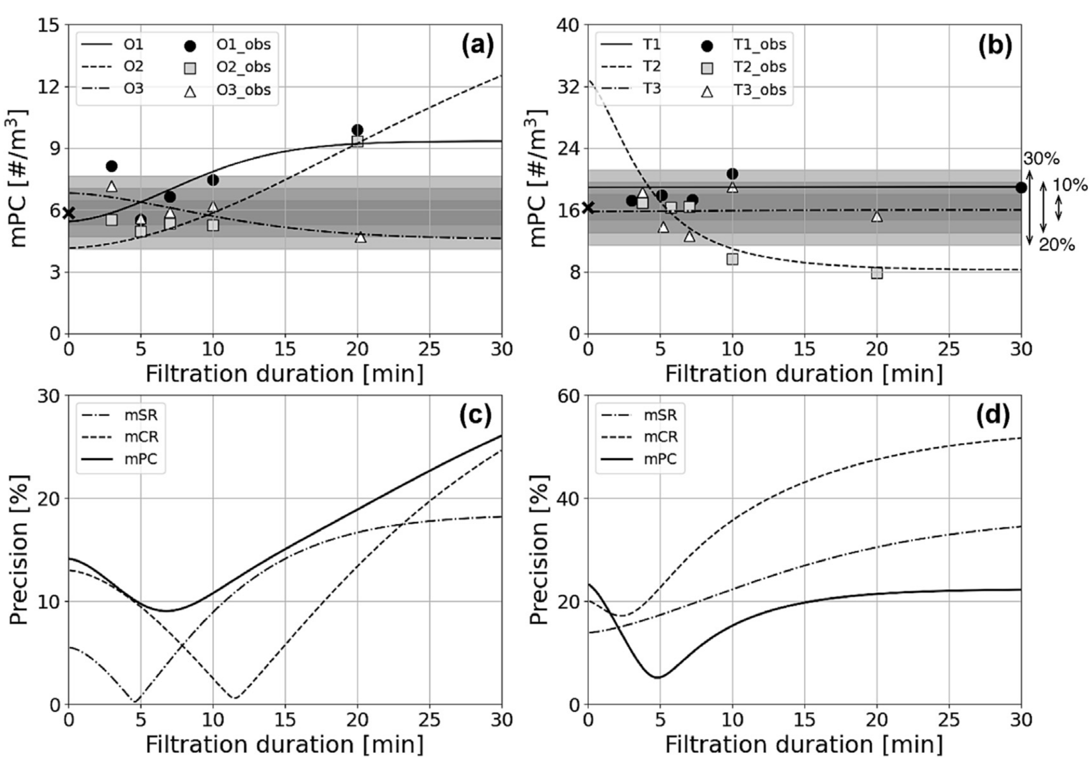

Fig. 4. Weibull distribution curves for mPC ((a) and (b)) compared to the observed mPC and mSR, mCR, and mPC precisions ((c) and (d)) in the Ohori (left side) and Tone-unga (right side) Rivers. In panels (a) and (b), the cross on the left vertical axis indicates the weighted mean mPC data in the 3-min, 5-min, and 7-min cases. The gray shades denote the domains with 10 % (dark gray), 20 % (middle gray), and 30 % (light gray) CV values based on the weighted mean, as shown on the right side of panel (b).

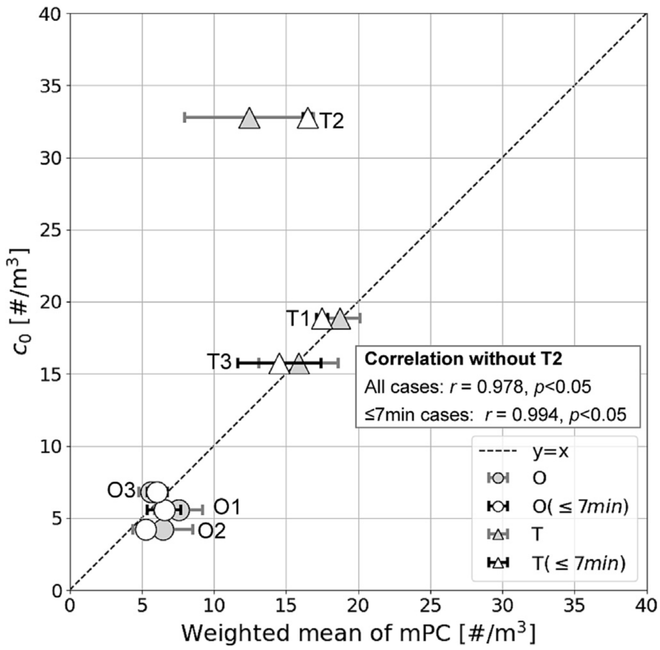

Fig. 5. Comparison between the weighted mPC mean (m_{cj}) and c_0. m_{cj} was calculated by using the mPC of all cases (white) and the short filtration cases (light gray). The error bar indicates the standard deviation (1σ). The broken line shows the $y = x$ line.

uncertainty should be determined by considering both plastic abundance in the water column and sample volume.

Furthermore, m_{cj} in the shorter-filtered cases (3-min, 5-min and 7-min cases) was in good agreement with c_0 of Eq. (14) except in T2 (Fig. 5). The difference between m_{cj} and c_0 in T2 must be induced by the overestimation of r_0 (Table 1). Thus, when discarding T2, the correlation between m_{cj} and c_0 in the shorter-filtered cases was 0.994 ($p < 0.01$), which is more strongly correlated than that in all cases ($r = 0.978, p < 0.01$). In addition, the calculated mSR, mCR and mPC precisions in the shorter-filtered cases were obviously lower than those in all cases (Fig. S6), supporting the concept that shorter-filtered sampling significantly reduces the mPC measurement uncertainty.

4.4. Strategy for deciding the filtration duration to obtain reliable data

The mPC uncertainty under net clogging was evaluated by approximating the growth in the sample volume and plastic abundance over time via the integral form of the WRF, and the uncertainty increased with increasing filtration duration. In contrast, a shorter filtration duration could result in mPC variance due to a low representativeness. Consequently, the filtration durations yielding the minimum mPC precision under the river conditions of the Ohori and Tone-unga rivers were 6.6 and 4.8 min, respectively. These filtration durations depended on both the plastic abundance and sample volume, but it could be difficult to obtain these factors in advance during field surveys. Our suggestion to determine a more appropriate filtration duration is to understand the decay in the filtration efficiency under actual river conditions.

Based on the definition of filtration efficiency (Eq. S1), it can be expressed by the following equation:

$$F(t) = F_0 \frac{\int_0^t q_0 \, exp\left[-\left(\frac{\tau}{\tau_q}\right)^{\alpha}\right] d\tau}{\int_0^t q_0 d\tau} = \frac{F_0}{t} \int_0^t \, exp\left[-\left(\frac{\tau}{\tau_q}\right)^{\alpha}\right] d\tau \qquad (17)$$

The decrease in the filtration efficiency can be obtained in Fig. 6. Smith et al. (1968) suggested that an efficiency higher than 85 % (gray line in Fig. 6) is needed to avoid net clogging effects. To date, 3–7-min filtration durations have often been selected in rivers (Bruge et al., 2020; Dris et al., 2018; Kataoka et al., 2019; Tanaka et al., 2022). In this sampling experiment, the filtration efficiencies at 3, 5, and 7 min are shown in Table 2. According to Smith et al. (1968), net clogging would have little effect on the plastic concentration evaluation until 3 min, while the plastic concentration measured in the 7-min sampling could be affected by clogging except at T1. Via extraction of the time sustained above an 85 % filtration efficiency in all packages from the generated filtration efficiency curves (sustained time (τ_s); Table 2), the sustained time varied between 2.6 and 6.2 min (3.2 and 7.1 min) throughout the experiment in the Ohori (Tone-unga) River, which was significantly correlated with τ_q ($r = 0.963$, $p < 0.01$). The above filtration duration range in the Ohori and Tone-unga Rivers permitted lower mPC uncertainties of 12 % and 9.5 %, respectively (Fig. 4). In the use of the net with $R = 2.41$, similar to our previous studies (Kataoka et al., 2019; Nihei et al.,

Table 2
Filtration efficiency and sustained time above 85 % efficiency.

	O1	O2	O3	T1	T2	T3
F_0	0.950	0.949	0.948	0.952	0.945	0.945
F at 3 min	0.837	0.896	0.918	0.921	0.885	0.864
F at 5 min	0.719	0.833	0.88	0.89	0.826	0.787
F at 7 min	0.604	0.76	0.834	0.854	0.763	0.708
Sustained time [min]	2.6	4.3	6.2	7.1	4.1	3.2

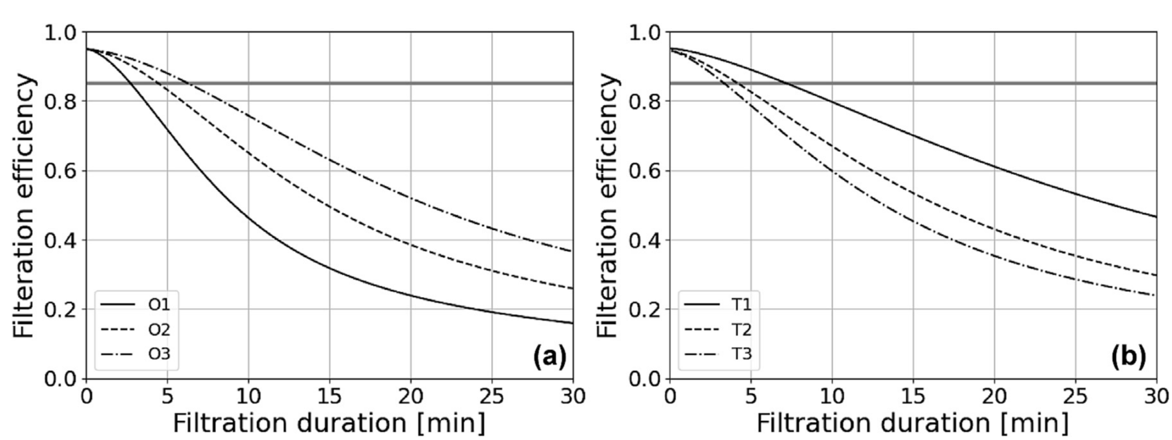

Fig. 6. Filtration efficiencies of the three packages in the Ohori (a) and Tone-unga (b) Rivers. The gray line in each panel indicates 85 % efficiency. The initial efficiency of each package was calculated according to Text S2.

2020), the selection of 5 min could be reasonable to reduce the uncertainty in mPC measurements.

After all, the determination of optimum filtration duration (i.e., sustained time) is equivalent to an estimation of τ_q in Eq. (17) because the former was linearly related to the latter as follows:

$$\tau_s = \beta_0 + \beta_1 \tau_q \left(r^2 = 0.941 \right) \tag{18}$$

where β_0 and β_1 are the intercept and slope, which are 0.116 ± 1.646 (95 % C.I.) and 0.420 ± 0.146 (95 % C.I.), respectively. Our suggestion is to conduct a preliminary survey to examine the decrease in filtration efficiency. The initial filtration efficiency (F_0) can be determined by the dimensions of the net and current velocities on the river surface, as shown in Text S2. The filtration efficiency decay at several filtration durations would be available by measuring the velocity inside and outside the net installed with two flowmeters, that is, $F = u'/u$ (Text S2). Therefore, α and τ_q would be determined by the nonlinear least square method, which can be made available upon request.

However, if the plastic contamination level was low, even a 5-min filtration would yield an insufficient sample volume, which would cause unexpected uncertainties. In this case, a net with a high open area ratio should be used. Smith et al. (1968) demonstrated that efficiencies above 85 % could be maintained even if the filtration duration exceeded 60 min by using a 0.333-mm mesh net with an open-area-ratio value of 6.4 (please refer to Fig. 2B of Smith et al. (1968). Thus, the use of a net with a high open area ratio could prevent the occurrence of net clogging even under long filtration durations. As a strategy to increase the open-area-ratio value, the shape and dimension of the net mouth or the surface area of the net gauze could be adjusted, e.g., by using a mouth-reducing conical net (Tranter and Smith, 1968). Our results emphasize the importance of describing the open-area-ratio value of nets used for microplastic sampling as well as their mesh size.

5. Conclusions

To clarify the effect of net clogging on the measurement of meso- and microplastic concentrations, the mPC uncertainty was evaluated based on its daily variation among different sites and filtration durations throughout a 1-day sampling experiment in small urban rivers (Ohori and Tone-unga Rivers). According to the basic mPC statistics for both rivers, the plastic contamination level in the Ohori River was significantly lower than that in the Tone-unga River, allowing us to examine the effect of net clogging in rivers with different contamination levels. Furthermore, the variances in both rivers consistently tended to increase with increasing filtration duration, suggesting that net clogging enhanced the mPC uncertainty. The temporal curves of the sample volume, plastic abundance, and mPC could be expressed by corresponding integral forms of the WRF. With the application of the WRF, the filtration durations yielding the lowest uncertainty (9.4 and 5.1) in the Ohori and Tone-unga Rivers could be estimated at 6.6 and 4.8 min, respectively, depending on the plastic abundance and sample volume. However, it could be difficult to obtain the plastic contamination level in advance. Thus, our suggestion is to predict the decay in the filtration efficiency over time by applying the WRF model. Based on a WRF-based filtration efficiency model, we found that the time sustained above 85 % filtration efficiency (sustained time) varied between 2.6 and 6.2 min (3.2 and 7.1 min) throughout the experiment in the Ohori (Tone-unga) River, which was significantly related to the scale parameter in the WRF model. The mPC uncertainty within this filtration-duration range varied between 12 % and 9.5 % in the Ohori and Tone-unga Rivers, respectively. Under a low plastic contamination level, a net with a high open area ratio should be used, which could prevent the occurrence of net clogging even under long filtration durations. These results emphasize the importance of describing the open-area-ratio value of nets used for sampling as well as their mesh size. Furthermore, this study contributes to standardizing the monitoring conditions of meso- and microplastics in aquatic environments during net sampling and to improving the reliability of the sampling results.

Data availability

Data will be made available on request.

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.

Acknowledgments

We are grateful to research assistants and students at Ehime University and Tokyo University of Science for their efforts in the laboratory analyses and field experiments. And we would like to thank American Journal Experts (https://www.aje.com/) for English language editing, and also thank anonymous reviewers for their comments to improve the manuscript. This work was supported by the Environment Research and Technology Development Fund (JPMEERF21S11900) of the Environmental Restoration and Conservation Agency of Japan, KAKENHI (21H01441), and a project (JPNP18016) commissioned by the New Energy and Industrial Technology Development Organization (NEDO).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scitotenv.2023.161942.

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