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► **To cite this version:**

Antoine Bruge, Marius Dhamelin court, Laurent Lanceleur, Mathilde Monperrus, Johnny Gasperi, et al.. A first estimation of uncertainties related to microplastic sampling in rivers. Science of the Total Environment, 2012, 718:137319, 10.1016/j.scitotenv.2020.137319 . hal-02481314

HAL Id: hal-02481314

<https://hal-enpc.archives-ouvertes.fr/hal-02481314>

Submitted on 30 Jun 2020

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1 A first estimation of uncertainties related to microplastic sampling in 2 rivers

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12

13 Abstract

14 Many studies have been conducted to quantify microplastic contamination, but only a few of
15 them have actually the sampling methodology and associated uncertainties. This study seeks
16 to examine the influence of sampling strategy on the confidence interval of river microplastic
17 estimates. 16 samples are collected in the Gave de Pau River (southwestern France) during a
18 three-hour window with a 330- μm mesh size net. Three different exposure times (3, 5 and 7
19 minutes) allow for a respective filtration rate by the net of 35.6 m^3 (3 samples), 59.4 m^3 (10
20 samples), and 83.2 m^3 (3 samples) of water. Organic matter contained in samples is removed
21 by hydrogen peroxide oxidation. The plastic particles are then counted and classified under a
22 binocular microscope. The microplastic concentrations vary between 2.64 and 4.24
23 microplastics/ m^3 , with a median value of 3.26 microplastics/ m^3 . Statistical analysis does not
24 show differences in microplastic concentrations for the three exposure times. This result

25 seems to demonstrate that a filtration of approx. 35 m³ of water is sufficient under similar
26 conditions (similar flow condition and degree of microplastic contamination) and can help
27 reduce sampling and sample processing time. Other analyses, based on 10 filtrations of 59.4
28 m³, show that the higher the number of samples, the lower the confidence interval. For
29 triplicates, the mean confidence interval reaches 15% of the median value. Thus, collecting
30 triplicates would seem to offer a reasonable optimum, in combining an acceptable error
31 percentage and time efficiency. These results might depend on the microplastic load of the
32 river, therefore making it necessary to conduct similar analyses on other rivers. This study
33 reports for the first time uncertainties related to microplastic sampling in rivers. Such findings
34 will serve to set up long term monitoring, highlight spatial differences between sites and
35 improve the accuracy of annual microplastic fluxes in rivers.

36

37 Keywords: microplastics, uncertainties, rivers, pollution, sampling methodology

38

39 Introduction

40 Over the last decade, considerable attention has been paid to plastic pollution in the marine
41 environment (Li et al., 2018; Li et al., 2016). Yet rivers, which are one of the major pathways
42 for plastics entering the ocean, have not been investigated to the same extent despite the fact
43 that in the past five years, an increasing number of studies have focused on river plastic
44 contamination. Data are still scarce and sampling methods need to be improved and
45 harmonised (Dris *et al.*, 2018; Eerkes-Medrano *et al.*, 2015; Wagner *et al.*, 2014). Moreover,
46 there is a major need to set up networks for measuring microplastics in inland waters to
47 monitor the evolution of their contamination over time and to set up databases at regional
48 scales, in line with regional regulations, like the Water Framework Directive in Europe

49 (although microplastics are not included as an indicator of good environmental status in its
50 current version).

51 Only easy to implement methodologies can be considered. However, they have to be
52 representative of the water bodies. The 200-year long experience in river monitoring suggests
53 that fluctuations in usual water quality parameters occur at much larger time scales than those
54 of sampling, and as a consequence, for instance, sampling 1 L of water is often enough to
55 measure both dissolved and particulate water quality parameters. Such an experience is
56 missing for microplastic contamination, in particular, for low concentration contamination,
57 taking into account that the dynamics of such particles in the water column is up to now badly
58 known. To the best of our knowledge, only two articles address small-scale temporal and
59 spatial variability of microplastics found in rivers (Dris *et al.*, 2018; Liedermann *et al.*, 2018).
60 Dris *et al.* (2018) analysed the temporal and spatial variability of fibre concentrations in the
61 Seine and Marne Rivers (France), using a 80 μm mesh net. They showed that the longer the
62 net deployment time, the lower the variability between consecutive samples. They also
63 assessed fibre distribution variability throughout the river cross-section and observed that
64 concentrations are similar across the water column and tend to increase near the banks.
65 Liedermann *et al.* (2018) studied microplastics distribution, using a 500 μm mesh net, within
66 the Danube River and also detected a slight tendency towards higher concentrations nearer the
67 banks.

68 On the basis of the sampling of 16 successive replicates and 3 sampling durations, in a river in
69 Southwestern France, we investigate the microplastic concentration fluctuations and assess
70 the corresponding uncertainties, and their variation with both the number of replicates and the
71 time exposure.

72 Materials and Methods

73 Study site and sampling methodology

74 This study was conducted on April 6th, 2018 between 11 am and 2 pm in the Gave de Pau
75 River, downstream of the Pau city centre yet still within the conurbation (Southwestern
76 France, lat.: 43.304828°, long.: -0.436492°). At this location, the river is 87 m wide with a
77 maximum depth of about 2 m. The flow is torrential with an annual mean discharge of 69.1
78 m³/s (average over the period February 2000 - July 2018 at station Q5231010 - Gave de Pau
79 at Artiguelouve - Pont de Lescar; *Banque hydro* database, 2018).

80 The river flow surface velocity was measured three times in a row before sampling using a
81 flowmeter (Flow Probe FR211) and ranged from 1.0 to 1.2 m/s. The average value (1.1 m/s)
82 was used to estimate the volume of water being filtered by the net. The mean river flow at
83 station Q5231010 was 75.8 m³/s (*Banque hydro* database, 2018).

84 Microplastic particles were sampled using a 330- μ m mesh size net with a rectangular opening
85 of 30 cm by 60 cm. This net was attached to a bridge roughly 6 meters from the river's left
86 bank. A second rope, fastened to the frame, was used to pull the net out of the water from the
87 bank. Two buoys were assembled on top of the frame to hold it just over the water surface,
88 and weights were used to keep it straight in the water column (Picture S1, Video S1).

89 A total of 16 samples were collected within a three-hour window. The maximum number of
90 samples that can be collected in this time frame ranges from 15 to 20 depending on the net
91 exposition time chosen. Beyond this time window, the hypothesis of steady state of the river
92 flow and pollution could be wrong. It was decided to use low net exposition times to avoid
93 clogging and reduce sampling and sample processing time. This is of high importance
94 considering that the method should be operational for large scale monitoring. The various
95 immersion times of the net chosen were 3 min (3 replicates), 5 min (10 replicates) and 7 min

96 (3 replicates), corresponding to 35.6, 59.4 and 83.2 m³ of filtered water, respectively. The 5-
97 minute net exposition time (59.4 m³) was thought to be the best option. This time was
98 therefore investigated further. Other values (i.e. 3 and 7 minutes, 35.6 and 83.2 m³
99 respectively) were also tested, although to a lower extent. Samples were stored in glass
100 containers with metal lids away from sunlight and at room temperature.

101

102 Microplastic extraction and identification

103 The samples were run through sieves with mesh sizes of 5 mm and 0.3 mm. The macroplastic
104 fraction (> 5 mm) was observed with the naked eye. The microplastic fraction, with a particle
105 size lying between 0.3 mm and 5 mm, was treated in order to remove organic matter (Masura
106 *et al.*, 2015; Hurley *et al.*, 2018). 20 mL of aqueous 0.05 M Fe(II) solution and 20 mL of 30%
107 hydrogen peroxide were added to a glass beaker containing the 0.3 to 5 mm fraction of the
108 sample. The resulting mixture was then placed on a lab bench at room temperature for 5
109 minutes before being heated to 75°C and held at that temperature until gas bubbles could be
110 observed. As the first bubbles cracked the surface, the beaker was removed from the hot plate
111 to avoid a violent reaction. When the solution had cooled slightly, the beaker was returned to
112 the hot plate and heated to 75°C for an additional 30 minutes. This operation was repeated
113 four times per sample due to the high quantity of organic matter.

114 The plastic particles were then counted and classified under a binocular microscope (Leica
115 EZ4) by colour (blue, red, transparent/white, black, green and other) and type (round,
116 fragments, angular and other shapes) (MERI, 2015). The fibres were not considered in this
117 study. The same operator handled all the samples. Tweezers were used to poke at individual
118 items whenever doubts arose.

119 Given the size range target (> 330 µm), and as only fragments were considered, the risk of
120 under or over estimation remains very low. In order to keep in mind the objective of a simple,

121 rapid and efficient method, it was decided not to proceed to a chemical characterisation, as
122 global methods based on Pyr-GC-MS are up to now high skilled and not quantitative, and
123 spectroscopic techniques (μ FTIR or μ Raman) are also high skilled and time consuming.

124

125 [Statistical analyses](#)

126 The number of plastic particles per sample was presented in terms of number of microplastics
127 per cubic meter (MPs/m³). The median microplastic concentrations for the three volumes of
128 filtered water, corresponding to the three exposure times, were compared using the non-
129 parametrical Kruskal-Wallis test and the R software (R Core Team, 2018). Since the number
130 of samples is small, non-parametric statistics, including medians and quantiles, are used.

131 Sampling uncertainties were assessed based on the 10 filtrations of 59.4 m³ (5 minutes). A
132 resampling technique was applied to a number of samples ranging from 3 to 10. The number
133 of existing combinations varied from 560 (for 3 samples) to 8,008 (for 10 samples), with a
134 maximum of 12,870 (for 8 samples). For each combination of samples, the mean medians and
135 mean standard deviations of microplastic densities per cubic meter were computed. As the
136 total number of combinations remains reasonable, all the combinations were tested. The
137 standard errors of the means and confidence intervals were then computed using a 95%
138 confidence level.

139 The delta of the standard errors of the means with respect to the median mean values has been
140 plotted vs. the number of samples in order to determine how the number of samples
141 influences the related uncertainties.

142

143 Results

144 In total, 3,191 microplastics were found within the 950 m³ of filtered water. 11.88 m³ of water
145 were filtered per minute of exposure. Exposure times of 3, 5 and 7 minutes allowed filtering
146 35.6, 59.4 and 83.2 m³ of water, respectively. No macroplastic was caught in the net.

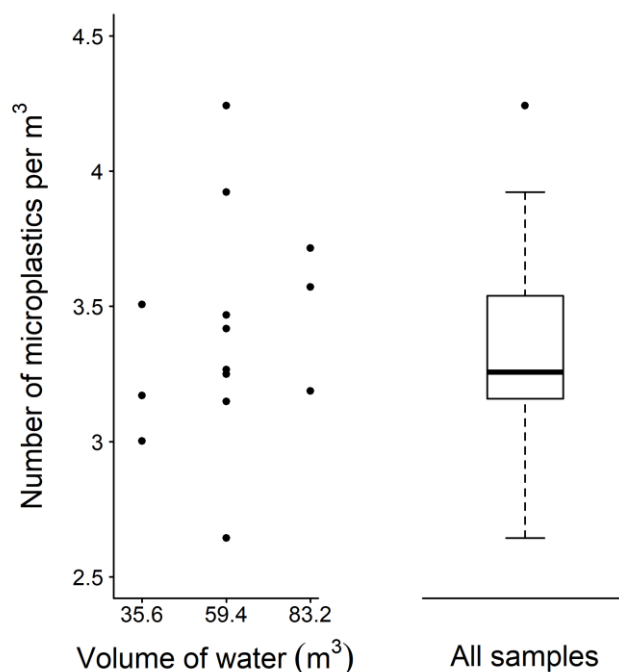
147 The microplastic concentrations varied between 2.64 and 4.24 MPs/m³, with a median value
148 of 3.26 MPs/m³.

149 The shapes and colours of microplastic particles are presented in Table S1. The microplastic
150 shape and colour variability between samples is low (the standard error ranges from 3% to 8%
151 for shape variability and from 0.3% to 9% for colour variability).

152

153 Influence of the net exposure time

154 The distribution of values recorded is shown in Figure 1.



155

156 Figure 1: Plot of the microplastics concentration vs. volume of filtrated water,
157 and boxplot of the microplastics concentration for all samples

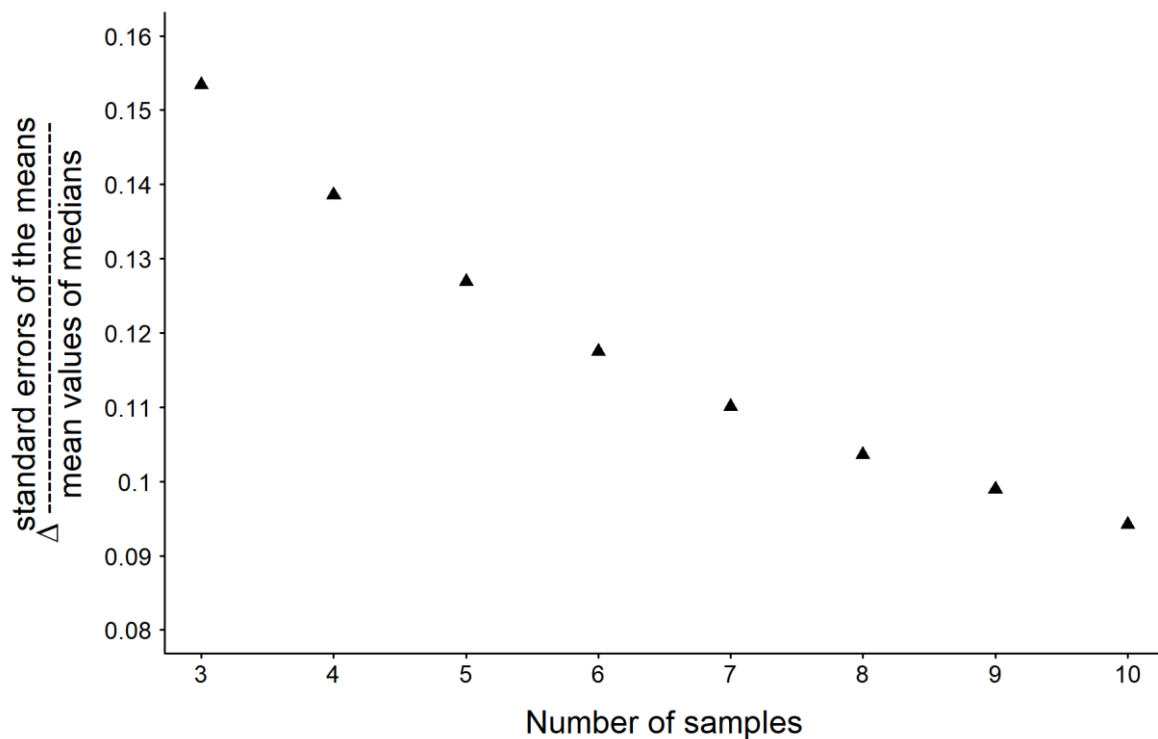
158

159 The microplastic concentration varies between 3.00 and 3.51 MP_s/m³, 2.64 and 4.24 MP_s/m³,
160 and 3.19 and 3.72 MP_s/m³ for the three exposure times (3, 5 and 7 minutes corresponding to
161 35.6, 59.4 and 83.2 m³), respectively. Despite the exposure time, the concentrations remain
162 close although the 5-minute exposure covers a wider range of values.

163 A Kruskal-Wallis test does not highlight any statistical differences between microplastics
164 concentration for each exposure time ($\alpha = 0.05$, $p = 0.5$). The median value of microplastic
165 density per cubic meter for the 16 samples equals: 3.26 ± 0.21 MP_s/m³.

166

167 **Uncertainties**



168

169 Figure 2: Delta of the standard errors of the means with respect to the median mean values vs.
170 the number of samples extracted

171

172 Figure 2 presents the delta of the standard errors of the means with respect to the median
173 mean values according to the number of samples extracted. This figure demonstrates that the

174 higher the number of samples are, the lower the confidence interval. All values are given in
175 Table S2.

176 Analyses conducted on the 10 filtrations of 59.4 m³ (5 minutes) have shown that for
177 triplicates, the mean confidence interval for the 560 combinations reaches 15% of the median
178 value, while the maximum confidence interval for the 560 combinations reaches 40% of the
179 median value. The confidence interval for all 10 samples is the lowest, i.e. reaching 9% of the
180 median value (95% confidence level). Values for mean and maximum confidence intervals for
181 each combination of samples are given in Table S2.

182

183 Discussion and conclusion

184 The median concentration of microplastics in the Gave de Pau River (3.26 ± 0.21 MPs/m³)
185 was ten times higher than concentrations measured in the Danube River between Vienna and
186 Bratislava (0.32 MPs/m³; Lechner *et al.*, 2014; exposition time and volume of water filtered
187 not mentioned) and in the Seine River downstream of the heavily-populated Paris Basin (0.35
188 MPs/m³, mesh size: 330 µm, net towed behind a motor boat for 15 minutes at about 2 m/s for
189 a volume of filtered water ranging from 182 to 200 m³, Dris *et al.*, 2015). Contrary to Dris *et*
190 *al.* (2018), who observed a decrease in variability between samples for longer exposure times,
191 our results do not present such a correlation. A comparison with other studies proves to be
192 difficult due to the significant differences in methods, extraction protocols and units (Li *et al.*,
193 2018). The work presented in this study is based on 16 samples, all extracted on the same day,
194 at the same location and within a time window for which the river flow and pollution can be
195 considered in a steady state. This value does not therefore reflect temporal variability in
196 microplastics concentration (van Emmerik *et al.*, 2018) and hence does not reflect the mean
197 annual load of the river either.

198 The large quantity of microplastics in our samples can be explained by the fact that two
199 former landfills, located near the riverbanks upstream of our site, were swept by the river
200 during several flooding events, the most recent occurring in February 2018. No chemical
201 characterisation was conducted. Visual identification of microplastics can sometimes lead to
202 substantial errors; however, given the size range target ($> 330 \mu\text{m}$), and because only
203 fragments were considered, the risk of under or over estimation remains very low. Moreover,
204 this bias is similar for all samples as the same operator handled all manipulations and it is
205 therefore possible to compare them. Microplastic concentrations for the three net exposition
206 times could not be shown to be statistically different. This result should be handled with
207 caution due to the low number in some groups of samples. This point in mind, it seems that
208 the filtration of approx. 35 m^3 of water is appropriate for future samplings under similar
209 conditions (similar flow condition and similar degree of microplastic contamination of the
210 river). This lower volume of water could reduce the time devoted to sampling, sample
211 treatment and identification. To verify that the conditions are similar to the ones describe is
212 this study, researchers are urged to use this method to investigate whether those results apply
213 to their rivers or not before starting a monitoring and setting up a monitoring station at a
214 specific river location. The number of samples taken for each volume of filtrated water should
215 ideally be the same to ensure statistics robustness. When choosing the different volumes of
216 water to be filtered as a first test, mind that a long exposure of the net might allow the catch of
217 rare particles, the setback being that the net could clog rapidly in case of high concentration of
218 suspended matter.

219 The 10 samples of 59.4 m^3 (5 minutes) extracted gave us the opportunity to study the
220 correlation between the number of samples collected and associated uncertainties. For
221 triplicates, the mean confidence interval reached 15% of the median value. The confidence
222 interval decreased quickly the higher the number of samples: it can drop to 9% of the median

223 value for all 10 samples. Thus, collecting triplicates seems to be a reasonable optimum that
224 combines an acceptable error percentage and time efficiency. These results might depend on
225 the microplastic load of the river, therefore prompting the need to conduct similar analyses on
226 other rivers. The order of magnitude of uncertainties reported in this paper can potentially be
227 affected by: i) time exposure, ii) river hydrodynamic conditions (low vs. high water levels),
228 and iii) the amount of microplastics sampled. As such, similar work must be performed under
229 other conditions.

230 This study has reported, for the very first time, uncertainties related to microplastic sampling
231 in rivers. The median concentration of microplastics in the Gave de Pau River recorded in this
232 study (3.26 ± 0.21 MPs/m³) is day-specific and do not allow for any extrapolation. Under
233 similar conditions, a 3-minute exposure time for the net (filtration of approx. 35 m³) and the
234 collection of triplicates seem to offer a reasonable optimum, by virtue of combining an
235 acceptable error percentage and time efficiency. Even if these results have to be confirmed by
236 increasing the number of samples per time exposure, this paper share a methodology that
237 helps design better microplastics studies. These results will help to set up long term
238 monitoring, determine microplastic fluxes and highlight the spatial difference between sites.

239

240 Acknowledgments

241 This study was funded by the French Ministry of Ecology and Solidary transition and the
242 Direction Inter-Régionale de la Mer Sud-Atlantique. The authors would also like to thank
243 Lauren Meshako from the Surfrider Foundation Europe and Robert Sachs for proofreading
244 this article.

245

246 Conflicts of Interest

247 The authors hereby declare the absence of any conflicts of interest.

248

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