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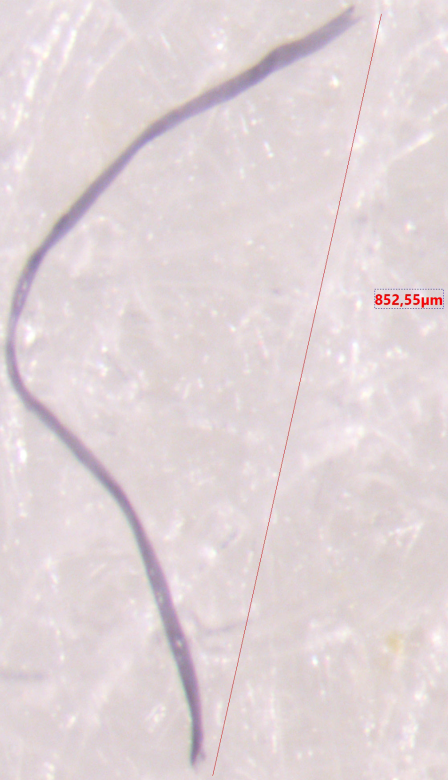

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MICROPLASTICS IN SEAFOOD FROM BELGIAN FISHERIES AREAS

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Bavo De Witte

Michael Dekimpe

Daphné Deloof

Kris Hostens

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Content

1 INTRODUCTION

Over the last decade, more than 400 papers have been published on the occurrence of microplastics in marine biota. The strong focus on microplastics in the marine environment can also be seen in food studies. Peer-reviewed A1-publications on microplastics occurrence in food are dominated by studies on seafood, followed by studies on drinking water and sea salt while little attention is paid to other, non-marine food matrices (EFSA, 2016, Robbens et al., 2021). Risks associated to microplastic contamination in food can be linked to direct toxicity from the uptake of small particles (Write & Kelly, 2017) or indirect toxicity, associated with the presence of chemical additives or pathogens on microplastics (OSPAR, 2017). This information has reached the general public through press releases, reports, events and awareness campaigns (Otero et al., 2021). Question raises to what extent the seafood consumer is exposed to microplastics and its associated risks. Many studies report on microplastic contamination in fish, including the gastro-intestinal tract (GIT) (EFSA, 2016). As the GIT is often not consumed, this data is not always relevant from a consumer perspective. For marine biota as well as humans, microplastics are considered too big to transfer from the GIT to the blood or lymph system (Write & Kelly, 2017; Lusher et al., 2017), which limits consumer exposure. Moreover, microplastic occurrence data in seafood may be subject to overestimation when appropriate background measures were not taken during analysis (Hermsen et al., 2017). Debate is also ongoing on the risks of microplastic exposure, as exposure to chemical or pathogens is not only influenced by the concentration of the chemical or biological pollutant but also by its bioavailability (EFSA, 2016: Koelmans et al., 2013). In this study, the occurrence of microplastics in seafood from Belgian fisheries is studied. Distinction in microplastic concentration is made between the consumed part of a seafood product and the non-consumed part. The main aim was to assess the exposure of the consumer of Belgian fisheries products to microplastics.

2 METHODS

2.1 SAMPLING

Based on relevance for Belgian fisheries, five fish species (sole (*Solea solea*), plaice (*Pleuronectes platessa*), brill (*Scophthalmus rhombus*), turbot (*Scophthalmus maximus*), cod (*Gadus morhua*)), brown shrimp (*Crangon crangon*) and edible crab (*Cancer pagurus*) were selected for the analysis of microplastics in fisheries products. Fish were sampled at five predefined Belgian fisheries areas: Southern North Sea (SNS), Central North Sea (CNS), English Channel (EC), Celtic Sea (CS) and Irish Sea (IS). The border between central North Sea and Southern North Sea was set at N52°36' as samples were taken within the Belgian beam trawl survey (BTS) and a clear distinction could be made between sample locations below and above this line. We chose to sample undersize fish, which is bycatch during fisheries surveys at the Belgian Demersal young fish survey (SNS, 2019), the Belgian BTS survey (CNS, SNS, 2019) and the English BTS survey (IS, 2019). Additional fish were sampled by ILVO seagoing observers in collaboration with commercial fishermen (EC, CS, 2019-2020). In some areas, not all fish species could be sampled (Table 1). For *S. rhombus* and *S. maximus* at SNS and CNS, insufficient fish were caught and additional GIT samples were taken on board during the campaign from undersized and sized fish (Table 1). Table 1 provides an overview of fish sampled per area, also indicating the ICES areas (Fig. 1) which correspond to each predefined area. *C. crangon* was sampled at the Southern North Sea. *C. Pangurus* at Central and Southern North Sea.

Table 1. Fish sampled for microplastic analysis. Sampling areas are based on ICES areas (Fig. 1).

Area	ICES area	Species	Total number of fish caught	Weight (g)	Length (cm)	GIT weight (g)
SNS	IVc	*S. solea*	29	53-114	19-23	1.1-5.4
		P. platessa	19	88-167	21-25	1.7-5.5
		S. rhombus	4	[1]	[1]	14.2-35.7
		S. maximus	5	[1]	[1]	7.4-43.1
		G. Morhua	6	176-533	32-36	12.4-25.6
CNS	IVb-IVc	*S. solea*	28	57-128	18-24	0.9-4.6
		P. platessa	18	98-169	21-25	2.4-7.4
		S. rhombus	10	[1]	[1]	2.4-19.0
		S. maximus	5	[1]	[1]	27.3-74.1
		G. Morhua	20	27-53	14-16	1.3-5.7
EC	VIId/VIIe	*S. solea*	28	71-145	21-25	1.4-7.0
		P. platessa	26	113-207	22-27	1.4-5.8
CS	VIIf/VIIg/VIIh	*S. solea*	35	73-121	21-25	1.6-3.4
		P. platessa	21	98-157	22-27	3.0-10.2
IS	VIIa	*S. solea*	22	91-132	21-24	1.2-5.3
		P. platessa	11	94-196	22-26	3.7-14.5
		S. rhombus	10	192-397	24-29	4.4-15.6
		G. Morhua	1	390	32	25.7

[1] Stomach dissection on board from fish >27 cm

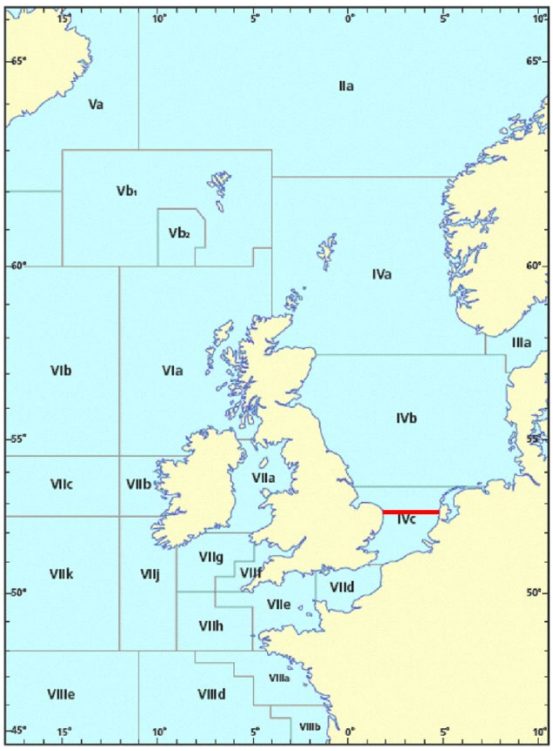

Figure 1. Separation of the Northwest Atlantic according to the ICES divisions (image © ICES) with in red, the line dividing CNS and SNS at N52°.36.

2.2 ANALYSIS PROTOCOLS

For all species, microplastic analysis was done on an edible part and a part that is not commonly consumed by European consumers. For fish, the filet was selected as edible part and the GIT as non-consumed part. For shrimps, analysis was done on the peeled tail muscle and on total shrimps. Crab meat was divided in the white edible meat within the claws and the brownish non-consumed inner organs of the crab.

Entire fish, shrimp and crab were stored on board at -20°C. Fish and crab were stored in a bag, shrimp were stored in a metal container. For samples of *S. rhombus* and *S. maximus* at ICES area IVb and IVc, no individuals were caught, and stomach samples were dissected on board and immediately wrapped in aluminium foil before storage at -20°C.

Fish and edible crab were dissected in a fume hood at the laboratory. Brown shrimp samples were also peeled in a fume hood. Fish samples did not include fish specimens with plastic or other materials in the mouth, nor everted or empty stomachs. All samples were stored at -20°C in metal containers after dissection/peeling. Samples were pooled to have approx. 20 - 30 g of sample (see Tables 2-5). For fish filet and the white meat of crab claws, a validated standard method was applied, able to analyse colored microplastics of 50 µm and larger and colorless microplastics of 200 µm and larger. A brief summary of main validation data is provided within annex 1. For fish GIT, edible crab GIT and shrimp, an additional density separation step was added to the standard protocol to separate dense particles from microplastics.

The analysis was initiated by a digestion with 100 ml 10% KOH / 10g sample for 24 h at 60°C. This digestion was done on a stirrer at a speed of 150 rpm. For the fish filet and the crab claws, the liquid sample was filtered directly after digestion through a paper filter (VWR 415) with particle retention of 12 - 15 µm and a pore size of 31 - 50 µm. For the fish GIT, crab GIT and shrimps, the particles in the samples were allowed to settle down for 24h at room temperature. The upper liquid layer was poured over a filter (VWR 310) with particle retention of 13 µm and a pore size of 25-60 µm. The remaining sample was brought into suspension again by 75 mL of a saturated sodium tungstate solution, prepared by dissolving 700 g of Na2WO4.2H2O in 1 L of demineralized water, followed by a settling phase of 24 h and decanting the liquid over the same filter (the density separation step was performed twice). After filtration, the filters were transferred to a petri dish and analyzed under a stereomicroscope (Leica M205), applying magnification factor 40 x. Particles were tentatively identified to be plastic by the hot needle test, i.e. by pointing a heated needle to the particle and evaluating if the particles melts or curls (Marine & Environmental Research Institute, 2015). Although indicative, validation showed that this technique allows differentiation between natural, semi-synthetic and synthetic fibres when performed by experienced lab technicians (annex 1).

All results were expressed in particles per 100 g of sample. For final results, the average blank signal, obtained from the procedure blanks, was subtracted. A limit of quantification (LOQ) was determined below which no quantification of particles in a sample could be done. The LOQ was calculated as three times the standard deviation of 18 procedure blank runs, analyzed in 2019 and 2020, and set at 1.8 particles per analysis.

2.3 QUALITY CONTROL

Stringent quality control measures were taken throughout the entire procedure. As background control measures, all materials were maximally covered. The equipment and devices used in the fume hood were daily cleaned with wet white cotton towels, and all glassware was rinsed 2 times with filtered water before use. Filtered water and solvents were prepared by filtration through a paper folding filter (VWR 310, particle retention 13 µm). Laboratory technicians wore white cotton clothing, a cotton white lab coat, and washed hands between each procedure step. No other lab members were allowed in the dedicated analysis area. Procedural blanks were checked daily, verifying the quality of the analysis and the contamination control measures taken. Air blanks were taken by placing petri dishes with wet paper filter open to the air during dissection. As a positive control, recovery tests with spiked particles were also included weekly.

3 RESULTS AND DISCUSSION

The results of the microplastic analysis in fish GIT (Table 2), fish filet (Table 3), *C. crangon* (Table 4) and *C. pagurus* (Table 5) are presented below. For each analysis, limit of quantification is equal to 1.8 particles per filter. Depending on the sample weight, this leads to a LOQ of 3.9 to 9.6 particles per 100g of sample.

Table 2. Microplastics observed in fish GIT of five different fish species, sampled at southern North Sea (SNS), central North Sea (CNS), English channel (EC), Celtic Sea (CS) and Irish Sea (IS).

Sample number	Species	Number of GIT /sample	Sampling area	Sample weight (g)	Number of particles per 100g
TUR 1	*S. maximus*	2	CNS	19.8	< LOQ
TUR 2	*S. maximus*	2	CNS	27.7	< LOQ
TUR 3	*S. maximus*	1	CNS	37.4	< LOQ
TUR 4	*S. maximus*	2	SNS	18.8	< LOQ
TUR 5	*S. maximus*	1	SNS	19.6	< LOQ
TUR 6	*S. maximus*	2	SNS	29.7	9.2
PLE 1	*P. platessa*	4	IS	28.4	< LOQ
PLE 2	*P. platessa*	3	IS	23.3	< LOQ
PLE 3	*P. platessa*	4	IS	31.1	< LOQ
PLE 4	*P. platessa*	6	CNS	21.0	< LOQ
PLE 5	*P. platessa*	6	CNS	27.6	< LOQ
PLE 6	*P. platessa*	6	CNS	22.3	< LOQ
PLE 7	*P. platessa*	7	SNS	24.5	< LOQ
PLE 8	*P. platessa*	6	SNS	24.2	< LOQ
PLE 9	*P. platessa*	6	SNS	24.9	15.1
PLE 10	*P. platessa*	8	EC	21.5	< LOQ
PLE 11	*P. platessa*	7	EC	22.3	< LOQ
PLE 12	*P. platessa*	8	EC	21.1	< LOQ
PLE 13	*P. platessa*	4	CS	24.5	< LOQ
PLE 14	*P. platessa*	5	CS	25.1	< LOQ
PLE 15	*P. platessa*	5	CS	25.7	< LOQ
SOL 1	*S. solea*	9	CNS	18.7	< LOQ
SOL 2	*S. solea*	10	CNS	23.4	< LOQ

SOL 3	*S. solea*	9	CNS	19.5	< LOQ
SOL 4	*S. solea*	10	SNS	26.6	< LOQ
SOL 5	*S. solea*	10	SNS	25.1	< LOQ
SOL 6	*S. solea*	9	SNS	28.5	9.6
SOL 7	*S. solea*	8	IS	26.2	< LOQ
SOL 8	*S. solea*	7	IS	21.6	< LOQ
SOL 9	*S. solea*	7	IS	27.8	< LOQ
SOL 10	*S. solea*	7	EC	21.7	< LOQ
SOL 11	*S. solea*	8	EC	22.8	< LOQ
SOL 12	*S. solea*	10	EC	21.6	< LOQ
SOL 13	*S. solea*	9	CS	21.4	< LOQ
SOL 14	*S. solea*	11	CS	23.8	< LOQ
SOL 15	*S. solea*	9	CS	22.0	< LOQ
BLL 1	*S. rhombus*	4	IS	23.2	< LOQ
BLL 2	*S. rhombus*	3	IS	27.6	< LOQ
BLL 3	*S. rhombus*	3	IS	35.4	< LOQ
BLL 4	*S. rhombus*	4	CNS	31.2	< LOQ
BLL 5	*S. rhombus*	4	CNS	25.7	< LOQ
BLL 6	*S. rhombus*	2	CNS	25.8	< LOQ
BLL 7	*S. rhombus*	1	SNS	34.7	< LOQ
BLL 8	*S. rhombus*	1	SNS	26.0	< LOQ
BLL 9	*S. rhombus*	2	SNS	30.5	< LOQ
COD 1	*G. morhua*	1	IS	25.7	< LOQ
COD 2	*G. morhua*	11	CNS	28.8	< LOQ
COD 3	*G. morhua*	10	CNS	24.8	< LOQ
COD 4	*G. morhua*	2	SNS	35.5	< LOQ
COD 5	*G. morhua*	2	SNS	37.7	< LOQ
COD 6	*G. morhua*	2	SNS	36.6	< LOQ

Table 3. Microplastics observed in fish filet of five different fish species, sampled at southern North Sea (SNS), central North Sea (CNS), English channel (EC), Celtic Sea (CS) and Irish Sea (IS).

Sample number	Species	Number of filet /sample	Sampling area	Sample weight (g)	Number of particles per 100g
PLE 1	*P. platessa*	1	IS	28.6	< LOQ
PLE 2	*P. platessa*	1	IS	23.7	< LOQ
PLE 3	*P. platessa*	1	IS	25.0	12.1
PLE 4	*P. platessa*	1	CNS	33.6	6.0
PLE 5	*P. platessa*	1	CNS	28.8	< LOQ
PLE 6	*P. platessa*	1	CNS	29.6	< LOQ
PLE 7	*P. platessa*	1	SNS	32.3	6.3
PLE 8	*P. platessa*	1	SNS	32.4	< LOQ
PLE 9	*P. platessa*	1	SNS	33.0	< LOQ
PLE 10	*P. platessa*	1	EC	32.3	< LOQ
PLE 11	*P. platessa*	1	EC	25.5	< LOQ
PLE 12	*P. platessa*	1	EC	25.5	< LOQ
PLE 13	*P. platessa*	1	CS	23.9	< LOQ

PLE 14	*P. platessa*	2	CS	24.4	< LOQ
PLE 15	*P. platessa*	1	CS	26.0	< LOQ
SOL 1	*S. solea*	2	CNS	27.7	< LOQ
SOL 2	*S. solea*	2	CNS	23.5	< LOQ
SOL 3	*S. solea*	2	CNS	26.9	< LOQ
SOL 4	*S. solea*	1	SNS	24.7	< LOQ
SOL 5	*S. solea*	1	SNS	30.5	< LOQ
SOL 6	*S. solea*	1	SNS	32.7	< LOQ
SOL 7	*S. solea*	2	IS	23.9	< LOQ
SOL 8	*S. solea*	2	IS	26.2	< LOQ
SOL 9	*S. solea*	1	IS	25.4	< LOQ
SOL 10	*S. solea*	1	EC	27.7	< LOQ
SOL 11	*S. solea*	1	EC	27.0	< LOQ
SOL 12	*S. solea*	1	EC	28.0	< LOQ
SOL 13	*S. solea*	1	CS	23.9	< LOQ
SOL 14	*S. solea*	1	CS	22.1	< LOQ
SOL 15	*S. solea*	1	CS	22.7	< LOQ
BLL 1	*S. rhombus*	1	IS	27.3	7.4
BLL 2	*S. rhombus*	1	IS	26.7	7.6
BLL 3	*S. rhombus*	1	IS	24.9	< LOQ
BLL 4	*S. rhombus*	1	CNS	33.0	< LOQ
BLL 5	*S. rhombus*	2	CNS	26.2	< LOQ
BLL 6	*S. rhombus*	2	CNS	32.9	< LOQ
COD 1	*G. morhua*	1	IS	29.0	< LOQ
COD 2	*G. morhua*	2	CNS	25.3	< LOQ
COD 3	*G. morhua*	2	CNS	24.0	< LOQ
COD 4	*G. morhua*	1	SNS	29.4	< LOQ
COD 5	*G. morhua*	1	SNS	25.1	< LOQ
COD 6	*G. morhua*	1	SNS	28.1	< LOQ

Table 4. Microplastics observed in brown shrimp, sampled at the Southern North Sea

Sample number	Species	Sample	Sample weight (g)	Number of particles per 100g
Shrimp P1	*C. crangon*	Peeled tail muscle	26.7	< LOQ
Shrimp P2	*C. crangon*	Peeled tail muscle	25.1	< LOQ
Shrimp P3	*C. crangon*	Peeled tail muscle	26.2	< LOQ
Shrimp U1	*C. crangon*	Unpeeled shrimps	20.0	< LOQ
Shrimp U2	*C. crangon*	Unpeeled shrimps	21.1	< LOQ
Shrimp U3	*C. crangon*	Unpeeled shrimps	22.4	< LOQ

Table 5. Microplastics observed in edible crab, sampled at the southern (SNS) and central North Sea (CNS).

Sample number	Species	Number of crabs /sample	Sample	Sampling area	Sample weight (g)	Number of particles per 100g
CRE C1	*C. pagurus*	1	Claw meat	CNS	46.5	< LOQ

CRE C2	*C. pagurus*	2	Claw meat	CNS	23.6	< LOQ
CRE C3	*C. pagurus*	1	Claw meat	CNS	26.2	< LOQ
CRE C4	*C. pagurus*	2	Claw meat	SNS	28.1	< LOQ
CRE C5	*C. pagurus*	1	Claw meat	SNS	28.8	< LOQ
CRE C6	*C. pagurus*	1	Claw meat	SNS	25.6	< LOQ
CRE G1	*C. pagurus*	1	GIT	CNS	32.4	< LOQ
CRE G2	*C. pagurus*	1	GIT	CNS	22.8	< LOQ
CRE G3	*C. pagurus*	1	GIT	CNS	25.8	< LOQ
CRE G4	*C. pagurus*	1	GIT	SNS	33.1	< LOQ
CRE G5	*C. pagurus*	1	GIT	SNS	24.6	< LOQ
CRE G6	*C. pagurus*	1	GIT	SNS	36.2	< LOQ

The number of particles detected in fish, brown shrimp and edible crab was low, as the number count did not exceed the LOQ for most samples. For fish GIT samples, only 3 out of 51 samples had a value higher than LOQ with a maximum of 15.1 particles/100 g in a sample of *P. platessa* of the southern North Sea. As this sample was a pooled sample of 6 fish GIT, average amount of particles per individual fish GIT in this sample was 0.6. For fish filet, only 5 out of 42 samples had a value above LOQ with a maximum of 12.1 particle per 100g. For brown shrimp and edible crab, no value higher than LOQ was recorded.

Since the number of microplastics larger than 50 µm is low in fish filet, crab meat and shrimp tail muscle, the consumption of Belgian fisheries products will not lead to a large exposure to microplastics of this size range. This is as expected, as microplastics of this size cannot cross epithelium membranes such as in the gastro-intestinal tract (Gouin, 2020; Lusher et al., 2017). Transfer of plastics through epithelium membranes is especially described for nanoplastics (1-100 nm) and small microplastics (<5-10 µm) (Abbasi et al., 2018; Avio et al., 2015; Collard et al., 2017; EFSA, 2016; Write & Kelly, 2017), but analytical methods to determine non-spiked nanoplastics in complex matrices such as fish GIT are currently still lacking.

Remarkably, the number of particles are also low in the GIT of fish. Many studies have reported on the presence of microplastic in fish, with a large variety in data, from <5% of fish (e.g. Foekema et al., 2013; Liboiron et al., 2016) to >90% of the fish with microplastics in the GIT (e.g. Jabeen et al., 2017). Differences between fish may result from feeding behavior, size, tropic level and ecological niche, but also geopgraphical differences may impact the amount of microplastics in fish GIT (Claessens et al., 2011; Rummel et al., 2016; Ory et al., 2017; Beer et al., 2018; Kühn et al., 2020). Next, also the quality of the analysis can play a big role. Many studies reporting high incidence lack appropriate quality control (Hermsen et al., 2017). The results of our study are in line with other studies on North Sea fish reporting low incidence of microplastics in the GIT such as Foekema et al., 2013 (2% of samples), Kühn et al., 2020 (1.8% of samples) and Hermsen et al., 2017 (0.25% of samples). Our results indicate that microplastics > 50 µm do no bioaccumulate in fish GIT. Due to the low number of microplastics found, no relevant comparison between species or areas could be done in this study.

In contrast to fish, only little data is available on microplastic occurrence in *C. crangon* and *C. pagurus*. Devriese et al. (2015) found on average 68 fibers per 100 g of brown shrimps from the English Channel and North Sea. The results of this study are, however, much lower. Korez et al. (2020) report strong depuration of particles from *C. crangon* within 48h. Similar to fish GIT, low microplastics numbers in *C. crangon* and *C. pagurus* suggest that microplastics do no bioaccumulate in this species.

4 Conclusions

The microplastic occurrence in seafood from Belgian fisheries was assessed, applying a validated method for microplastic analysis. Colored microplastics >50 µm and colorless microplastics >200 µm could be determined by the applied method. In most of the fish samples, for GIT as well as filet, values did not exceed the limit of quantification. Also in samples of *C. crangon* and *C. pangurus*, limit of quantification was not exceeded. These results indicate that microplastics do no bioaccumulate in fish, brown shrimp and edible crab. The exposure to microplastic by consuming seafood from Belgian fisheries products is limited.

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7 ANNEX 1: SUMMARY OF METHOD VALIDATION DATA

For method validation, water and mussel tissue was spiked with red polystyrene beads (size 106-125 µm and 500-600 µm) and colorless polyethylene beads (size 106-125 µm and 500-600 µm). For each type and size, spiking was done with 10 and 40 particles per sample. Validation was done by 2 lab technicians on 6 days with 2 independent analyses per sample per day.

Accuracy (%) was determined as: 100 + ((X_{av} - C_{ref})/C_{ref} * 100) with X_{av} the average measured value and C_{ref} the spiked amount of reference particles. Precision was determined as CV_R (%) = S_r/X_{av}*100 with S_r the standard deviation of the measured value.

As can be seen in the tables below, predefined quality criteria were met for colored particles as well as colorless particles of 600 µm. As criteria were not met for colorless particles of 100 µm, accuracy and precision analyses was also done on colorless particles of 200 µm (2 days, 2 independent measurements/day). For 200 µm colorless beads, criteria were met with an accuracy of 87.5% and a precision of 9.5%.

Table A1. Accuracy of the method, applying spiked water and mussel samples.

Beads		Matrix	X_{av} (#)	C_{ref} (#)	Ac. (%)	b (%)	± 20%
600 µm	Colorless	Water	9.8	10	97.5	-2.5	OK
		Mussel	9.3	10	92.5	-7.5	OK
	Red	Water	10.0	10	100.0	0.0	OK
		Mussel	9.8	10	98.3	-1.7	OK
	Colorless	Water	38.2	40	95.4	-4.6	OK
		Mussel	36.7	40	91.7	-8.3	OK
	Red	Water	39.8	40	99.4	-0.6	OK
		Mussel	39.9	40	99.8	-0.2	OK
Beads		Matrix	X_{av} (#)	C_{ref} (#)	Ac. (%)	b (%)	± 30%
100 µm	Colorless	Water	3.3	10	33.3	-66.7	NOK
		Mussel	3.7	10	36.7	-63.3	NOK
	Red	Water	8.5	10	85.0	-15.0	OK
		Mussel	8.6	10	85.8	-14.2	OK
	Colorless	Water	19.0	40	47.5	-52.5	NOK
		Mussel	18.8	40	46.9	-53.1	NOK
	Red	Water	36.4	40	91.0	-9.0	OK
		Mussel	32.6	40	81.5	-18.5	OK

Table A2. Precision of the method, applying spiked water and mussel samples.

Beads		Matrix	C_{ref} (#)	X_{av} (#)	S_R (#)	CV_R (%)		± 20%
600 µm	Colorless	Water	10	9.8	0.5	4.6	6.7	OK
			40	38.2	3.4	8.9		
		Mussel	10	9.3	1.0	10.4	10.0	OK
			40	36.7	3.5	9.5		
	Red	Water	10	10.0	0.0	0.0	0.8	OK
			40	39.8	0.6	1.6		
		Mussel	10	9.8	0.4	4.0	2.3	OK
			40	39.9	0.3	0.7		
100 µm	Colorless	Water	10	3.3	2.4	71.6	53.8	NOK
			40	19.0	6.8	36.0		
		Mussel	10	3.7	1.8	49.8	43.5	NOK
			40	18.8	7.0	37.2		
	Red	Water	10	8.5	1.3	15.5	13.6	OK
			40	36.4	4.3	11.7		
		Mussel	10	8.6	1.3	15.3	17.6	OK
			40	32.6	6.5	19.8		

Next to accuracy and precision, robustness and specificity of the method was evaluated within validation and a limit of quantification was determined, based on the analyses of procedure blanks.

Within specificity tests, hot needle test proved reliable in making distinction between synthetic polyester and polyacryl fibres compared to semi-synthetic rayon and natural cotton, jute, linen and wool.

Contact

Bavo De Witte, Scientific Researcher
Flanders Research Institute for Agriculture, Fisheries and Food
Animal Sciences Unit
Aquatic Environment and Quality
Ankerstraat 1
8400 Oostende - Belgium
T + 32 59 56 98 64
bavo.dewitte@ilvo.vlaanderen.be

ILVO

ILVO

Flanders Research Institute for Agriculture, Fisheries and Food
Burg. Van Gansberghelaan 92
9820 Merelbeke - Belgium

T +32 9 272 25 00
ilvo@ilvo.vlaanderen.be
www.ilvo.vlaanderen.be