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BRIEF REPORT

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Survey among European and Canadian feed control units on monitoring packaging material residues in feed by microscopy analyses

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ABSTRACT

Macro- and microscopic evaluation of feed includes detection of animal proteins, botanical ingredients and impurities, and prohibited ingredients such as packaging material (PM), according to Regulation (EC) 767/2009. In addition, detection of micro-plastics (possible degradation products of some of the PM) is getting attention. PM can harm animals or disturb their feed intake, pollute the environment, and are considered as undesired impurities in feeds. These materials do not consist of a definite molecule, group of molecules, living species or definite bodies. They can be plastic foil, hard plastic, metal pieces, paper, wood or some combination of materials. Their features (sharp, pointed) can be as important as the material itself. This is a typical topic for microscopy detection and evaluation. This short review presents the work done on detection of PM in 15 monitoring entities (institute, laboratories). Since 2011, some institutes have analysed more than 20 samples each year and the incidence of non-compliant samples will be presented here. Thirteen out of 15 entities have an active monitoring, whereas others have passive surveillance (done while performing other microscopy analyses). The protocols used by the different entities depend on sample types and analysts, highlighting a need for harmonisation.

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Former food products; packaging material; limit of tolerance

HIGHLIGHTS

- Former food products as ingredients for animal feed reduce food losses but contain residues of packaging material (PM).
- The microscopic examination and evaluation of feeds contribute to the safety of ingredients issued from food re-cycling and by-products valorisation.
- The lack of a prescribed method and limit of tolerance for PM cause variability in survey results.

Introduction

Intensive livestock breeding is in the fire of criticism. Pressure from the population in general and especially from vegans and anti-speciesist who want to completely abolish meat and other animal proteins production and consumption is increasing steadily. Among arguments against animal breading, the cruelty of exploiting any kind of animals, the very poor conditions of keeping, fattening and slaughtering livestock, also arguments about the high carbon-footprint of animal feeding production are presented (Mota-Rojas et al. 2023).

Former food products (FFPs) are defined as foodstuffs, other than catering reflux, which were manufactured for human consumption in full compliance with the food law, but which are no longer intended for

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human consumption. This may be due to logistical reasons or to defects in manufacturing or packaging. The food content does not present any health risk when used as feed (Commission Regulation (EU) 2017). By using a maximum of by-products and recycled materials in animal feed, a balanced agriculture and food production – including animal protein for humans (meat, milk, egg) – could still be sustainable and better accepted (Luciano et al. 2022; Nath et al. 2023). From a circular economy perspective, by using FFP, it is possible to reduce food losses since these ingredients are suitable for animal feed, especially for pigs, poultry and young animals (Luciano et al. 2020; Pinotti et al. 2021; James et al. 2022). However, residues of packaging materials (PMs) can still be present after unpacking and processing (Tretola et al. 2017; van Raamsdonk et al. 2022).

PM fragments might harm animals or disturb their feed intake and may even pollute the environment (Zolotova et al. 2022). The toxicity of dyestuff, for example, is under study. Overall, these materials do not consist of one definite molecule, group of molecules or a defined body. They can be plastic foil, hard plastic, metal pieces, paper, wood or some combination of materials. Their features (sharp, pointed) can be as important, considering the undesired aspect, as the material itself (van Raamsdonk et al., 2011).

The microscopic examination and evaluation of feeds and feed materials contribute to the safety of ingredients issued from food re-cycling and by-products valorisation by:

a. proving the identity and purity of feed materials,
b. establishing the presence of impurities or prohibited contaminants,
c. recognising undesired elements or plagues, such as invading insects and moulds,
d. estimating the share of the recognisable ingredients,
e. contributing to the traceability of ingredients and contaminants.

In contrast to the instrumental analysis of analytic chemistry, the visual identification of a large diverse range of materials relies heavily on the knowledge of biology and adjacent disciplines (van Raamsdonk, Frick, et al. 2023; van Raamsdonk, Smits, et al. 2023). Also, the knowledge of the processes involved in food and feed production is important, giving information on the type of particles that may be found (crushed, extruded, spray-dried, etc.). The analyst can gain routine in a day-by-day process of investigating sample material, which is a long and intensive process. Sources for expertise can be found among handbooks, training by experienced scientists, collections of reference materials and images, and dedicated expert systems on a computer. Guidance from supervisors with experience in the specific requirements of visual and microscopic investigations is strongly recommended.

Considering that PM was relatively new for microscopists, and, in most cases, FFP (mostly cooked and baked food), as well as PM have little histological structures, training and proficiency testing were part of the task program of the IAG (International Association for Feedingstuff Analysis – Section Feedingstuff Microscopy), which gather food and feed microscopists all over the world.

van Raamsdonk also discussed the aspects of quantification and intervention limits, as well as the obvious link to the method of detection: Regulation (EC) 767/2009 Annex III (Commission Regulation (EC) 2002) mentions zero tolerance for the presence of remnants of PM in FFPs. In practice, relatively low action limits were installed in member states (van Raamsdonk et al. 2011). Several methods for the detection of PM have been developed, with the scope of bakery by-products (van Raamsdonk et al. 2012) or compound feed containing bakery by-products (Amato et al. 2017). Later, candy syrups were added to the scope (van Raamsdonk et al. 2022).

PM residues in animal feed have been a concern since more than 10 years among the IAG members and subject of several presentations at annual meetings of the association, as well as in conferences such as the 7th Feed Conference: Feed 2021 in Vienna, Austria. Training, proficiency testing and protocol harmonisation were comprehensively discussed over the years of activity of this expert group. In 2021, 29 analysts participated in a proficiency test organised by the IAG and demonstrated the usefulness of the microscopy methods in this field (van Raamsdonk et al. 2022).

In 2023, members of the IAG decided to gather data allowing to produce and present an overview of how well implemented is the monitoring on PM residues in Europe and getting insight in the evaluation of the situation according to the presence of such residues in animal feed.

The aim of the study was:

a. to grasp how widespread and active the surveillance of PM was in Europe,
b. to list the methods used and protocols adopted,
c. to discuss the possible harmonisation of the limit of tolerance (LOT),

d. to evaluate the proportion of contaminated samples.

In June 2023, a questionnaire was sent to IAG members and one other official laboratory (IZSTO, Italy) with the aim of recovering information on whether they were performing a monitoring on the presence of PM residues in feed, and if yes, since when. Questions were asked on the number of analysed samples, FFP or mixed feed, method used, LOT applied, number of samples below and above LOT. The last question was about the willingness to participate in the publication of a compilation of data on the subject.

To the best of our knowledge, our survey presents the first results of content of PM in animal feed from the industry in Europe and Canada.

Materials and methods

Fifteen laboratories participated in the survey: six from Germany and one laboratory each from Austria, Switzerland, Croatia, Belgium, Denmark, Italy, Sweden and Slovenia. A Canadian laboratory was the only one outside of Europe. The survey was not announced in advance which had the consequence that the data collected was not registered in the same way in all institute. We decided to ask for information on samples of FFP on the one side (samples from bakery and candy industry) and on all other mixed feed on the other side.

List of participants:

AGES: Austrian Agency for Health and Food Security, Austria;
Agroscope: Switzerland;
CVI: Croatian Veterinary Institute, Croatia;
CVUA: Chemical and Veterinary Investigation Office, Germany;
FAVV: Federal Agency for the Safety of the Food Chain, Belgium;
FVST: Danish Veterinary and Food Administration, Denmark;
IZSTO: Experimental Zooprophylactic Institute of Piedmont, Italy;
LAVES: Lower Saxony State Office for Consumer Protection and Food Safety, Germany;
LLG: State Institute for Agriculture and Horticulture, Sachsen-Anstalt, Germany;
LUFA Nord-West: Agricultural examination and research institute, Germany;
LUFA Rostock: Agricultural examination and research institute, Germany;
SGS, Germany;
SVA, Swedish Veterinary Agency, Sweden;
UL VF: University of Ljubljana, Institute of Food Safety, Feed and Environment, Slovenia;
CFIA: Canadian Food Inspection Agency, Canada.

Methods

The general protocol is based on sieving the sample material, manual selection of particles that match the types of PMs (paper, board, plastic, clips, wires, etc.) from the appropriate fractions, defatting and dehydration of the selected material, weighing and calculation of the percentage (w/w). Knowledge of all the possible types of particles present in the PM and their properties is essential. Cleaning (mainly defatting) is important in the case of paper or cardboard fragments and of quantitative results, but not when applying a zero tolerance for the LOT. Furthermore, solvents can damage certain plastics but are performant to separate metal pieces. Specificity of used methods are presented in Table 1.

The methods declared to be used are the following: the WFSR (Wageningen Food Safety Research) method (van Raamsdonk et al. 2012, 2022); the VDLUFA (Association of German Agricultural Analytic and Research Institutes) 30.9 method (VDLUFA 2021); and in-house methods modified from the WFSR methods and the VDLUFA method. One of the in-house methods is described in the study by Amato et al. (2017). These methods are highly comparable, and the small differences reflect the purpose of the analyst. It is possible to use one protocol for screening and the more complete protocol for confirmatory and quantification purposes (Table 1).

In the original WFSR method, which is the more complete, approximately 500 g of sample without pretreatments is weighted and analysed. The sample is sieved at mesh sizes of 2 and 1 mm. In in-house methods and in VD LUFA method, a sieve with a mesh size of 0.5 mm can additionally be used. The two largest fractions (>2 mm and 1–2 mm) are weighed and investigated for remnants of presumed PMs. All particles that are not native to the matrix are picked up by a pair of tweezers and kept. If necessary, a magnifying glass (between 5× and 50× magnification; in-house method magnification up to 400× for small pieces of PM) can be used. It is impossible to isolate all target particles from the finest fraction, which is, consequently, only screened.

Table 1. Specificity of used methods.

Method	WFSR	VD LUFA	In-house method
Weight of samples	500 g	At least 250 g	500 g
Sieves	1 mm and 2 mm	0.5 mm	0.5–2 mm
Solvents use	Obligation	Optional	Optional
	Water, TCE	Water, TCE, acetone, petroleum ether	Water, TCE, chloroform
Magnification	5×–50×	5×–50×	5×–400×
Weight of determined PM	Obligation	Optional (if LOT applicable)	Optional (if LOT applicable)

WFSR: Wageningen Food Safety Research VDLUFA is for: Verband Deutscher Landwirtschaftlicher Untersuchungs- und ForschungsAnstalten.

When a quantitative result is needed, the selected materials are weighted and exposed to defatting in a baker with 50 mL of tetrachloroethylene (TCE) for 10 min. The TCE is decanted over a sieve of the appropriate mesh size. In in-house methods and VD LUFA method, other solvents for defatting and cleaning (such as chloroform, acetone, petroleum ether and water) were used. The remnants are placed in a sieve for drying overnight. For final dehydration, remnants are placed in an oven at 60 °C for 4 h. Results are expressed in net weight and percentage. If desired the nature of the remnants found can be established (e.g. paper, plastic, aluminium foil, etc.).

If the authority applies zero tolerance (LOT is equal to the limit of detection), then the method is purely qualitative and defatting as well as dehydration are usually not performed.

Results and discussion

The presented study shows contribution of the field of feed microscopy to safe, cheap and ecological feed production. Besides the knowledge of numerous and varying ingredients such as food by-products (oil cakes, fish and meat and bone meal, oyster-shell, glutens, wheat bran, beet pulp, fruit pomaces, brewers' and distillers' residues, etc.) knowledge on 'recycling material' such as bakery-by-products and FFP in general was gained over the last decades.

The collected results in the frame of PM detection in feed monitoring are presented in Table 2. Ten out of 15 laboratories which participated in the survey analysed both type of samples, FFP and mixed feed. In the period from 2011 to 2023, 2437 FFP samples and 4433 samples of mixed feed were analysed by the European laboratories surveyed. Most laboratories declared metal, plastic, paper and wood as PM. A few reported also glass and stone. Some types of PM selected from the analysed samples are shown in Figure 1.

In Europe, non-compliant (positive above LOT) FFP samples were 12.2%, and non-compliant mixed feed samples 2.8%, showing -on top of the possibility that not all mixed feed contain FFP- a probable dilution effect in the mixed feed, where FFP is only one of several ingredients.

In laboratories which applied LOT (0.01% for CVUA, SGS and SVA; 0.05% for IZSTO; 0.125% for FAVV and 0.2% for LLG, LUFA Nord-West and LUFA Rostock) another 18.4% samples of FFP and 9.1% mixed feed were positive as well, but below LOT.

In the same period in CFIA (Ottawa, Canada), 84 samples of FFP were analysed by the laboratory surveyed, among which 23% samples were positive below LOT (0.1%) and 28.6% samples were positive above LOT. No mixed feed samples were analysed by CFIA.

Similarly, FVST analysed only FFP, but applied a zero tolerance; their percentage of positive samples was 45.0%.

On the contrary, in UL VF and SVA, 1086 of mixed feed (1063 and 23 samples, respectively) were analysed and no FFP. UL VF had no positive samples and SVA registered two positive samples, both above LOT.

LUFA-Rostock (DE) did not separate samples of FFP and mixed feeds; therefore, their results are in italic in the mixed feed section. A large number of samples were analysed, with a relatively high percentage of positive ones (40.3% below and 1.4% above LOT).

In Agroscope (CH) and UNI VF (SI), samples are not analysed strictly for PM but in the frame of other microscopic examination (total number not recorded by Agroscope). At Agroscope, the results for the FFP presented in Table 1 are mostly confirmatory analyses performed on single ingredients of suspect mixed feed.

It is interesting to note that laboratories that have analysed PM in FFP and mixed feed over a shorter period of time have analysed more samples than laboratories with a longer study period of examination. AGES has analysed a total of 1918 samples since 2015 (240 samples per year), and LUFA Nord-West a total of 981 samples since 2017 (164 samples per year). In contrast, Agroscope has analysed a total of 14 samples since 2011 (1.17 samples per year), CFIA a total of 84 samples since 2010 (6.5 samples per year), and CVI a total of 149 samples since 2013 (14.9 samples per year). FAVV and LAVES present long-term records with

Table 2. Collected results of the survey of monitoring packaging material in some European countries and Canada.

				FFP									Mixed feed												
					Positive				Material					Positive				Material							
	Institution	Used method	Tolerance (up to %)	*N*	$<$LOT	%	$>$LOT	%	Metal	Plastic	Paper	Other	*N*	$<$LOT	%	$>$LOT	%	Metal	Plastic	Paper	Other	Year of start of analyses	Targeted	Random	Official control
1	AGES (Austria)	VDLUFA 30.7/30.9	0	909		0.0	66	7.3	yes	yes		Glass	1009		0.0	16	1.6	yes	yes		Stone	2015	yes		yes
2	Agroscope (Switzerland)	WFSR	0	14		0.0	4	28.6	yes	yes	yes	Wood										2011	yes	yes	yes
3	CVI (Croatia)	WFSR	0	14		0.0	1	7.1	yes	yes	yes	Ruber	135		0.0	2	1.5				Wood	2013	yes		yes
4	CVUA (Germany)	WFSR	0.01	73		0.0	24	32.9	yes	yes	yes		225	44	19.6		0.0	yes	yes	yes	Wood	2017	yes		yes
5	FAVV (Belgium)	WFSR	0.125	502	156	31.1	4	0.8	yes	yes	yes	Stone, wood, tissue fibre	4	4	100.0		0.0		yes	yes	Rope	2011	yes		yes
6	FVST (Denmark)	WFSR	0	40		0.0	18	45.0	yes	yes	yes											2017	yes		yes
7	IZSTO (Italy)	In house	0.05	5		0.0		0.0					279		0.0	14	5.0	yes	yes	yes		2016	yes		yes
8	LAVES (Germany)	In house	0	188		0.0	173	92.0	yes	yes	yes	Wood, glass	290		0.0	76	26.2	yes	yes	yes	Wood	2012	yes		yes
9	LLG (Germany)	VDLUFA 30.7/30.9	0.2	33	28	84.8	2	6.1		yes	yes		48		0.0		0.0					2019	yes		yes
10	LUFA Nord-West (Germany)	In house	0.2	496	123	24.8	5	1.0	yes	yes	yes	Ruber	485	14	2.9	4	0.8	yes	yes	yes		2017	yes		
11	LUFA Rostock (Germany)	VDLUFA 30.7/30.9	0,2										*843*	*340*	*40.3*	*12*	*1.4*		*yes*	*yes*	*yes*	2016	yes		
12	SGS (Germany)	In house	0.01	163	143	87.7		0.0	yes	yes	yes		29	1	3.4		0.0			yes	yes	2015	yes		
13	SVA (Sweden)	WFSR	0.01										23		0.0	2	8.7		yes			2016	yes		
14	UL VF (Slovenia)	N/A											1063		0.0		0.0					2013		yes	
Total				2437	450	18.4	297	12.2					4433	403	9.1	126	2.8								
	CFIA (Canada)	In house	0.1	84	20	23.8	24	28.6	yes	yes	yes	Glass										2010	yes		yes

N: number of samples; LOT: limit of tolerance; FFP: formed food products.
Italicized values show laboratory sent gathered results of FFPs and mixed feed.

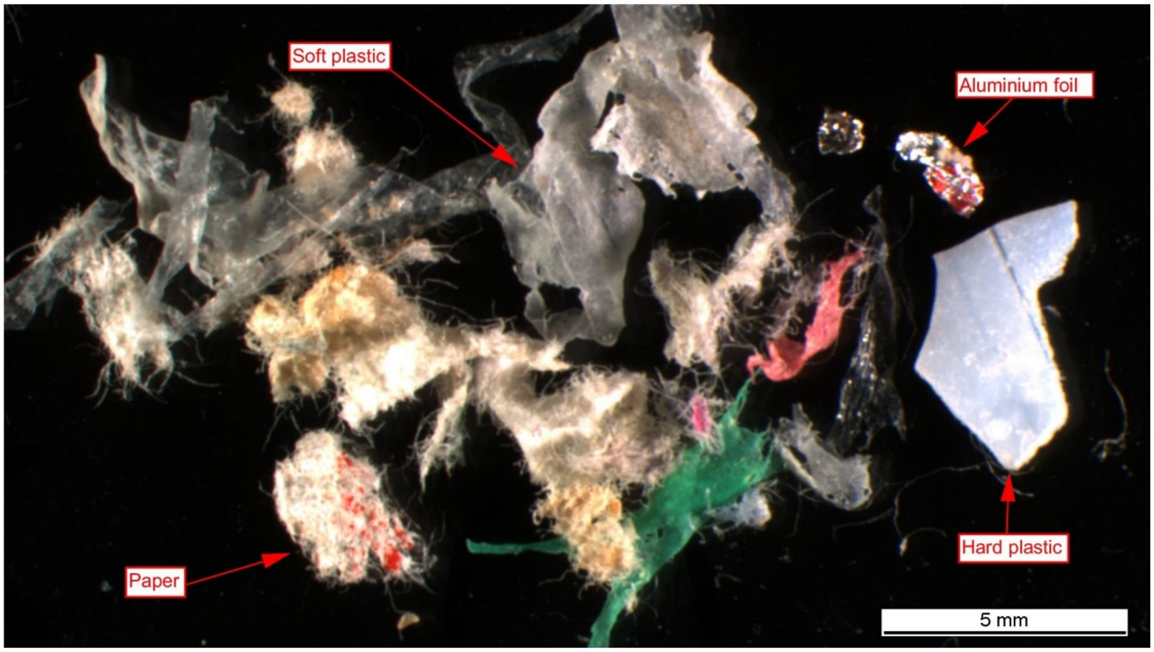

Figure 1. Packaging material residues collected by a microscopist from a sample of former food products intended for animal feed.

intermediate sampling rate: a total of 506 samples since 2011 (42.2 samples per year) for FAVV and 478 samples since 2012 (43.5 samples per year) for LAVES. IZSTO started the dedicated survey more recently (2016), but with a fair number of samples (285 total which means 40.7 per year). The time-course and intensity of sampling for each institute is not detailed in this presentation, and such particularities could be due to the fact that FFP is not used in all countries as frequently as in Germany, Belgium and Austria. In these countries, passive surveillance has probably been recognised as not sufficient, and consequently dedicated and specific sampling increased or started at different time-points in the last years.

The incidence of non-compliant samples, regarding PM, logically also varies according to the countries (availability and good implementation of the ingredient FFP in practice). In entities with many samples analysed yearly, the percentage of non-compliant samples also varies depending on LOT applied. The general tendency is a relatively high incidence among FFP samples: 34.4% for all samples together (791 positive from 2521), – showing up to 92% when applying the zero tolerance (e.g. at LAVES) – and a recurring but lower presence in mixed feed samples: 11.9% for all samples together (529 from 4433 samples), with a maximum of 26% (LAVES) when applying zero tolerance. An example of a low percentage of non-complying samples correlated with a LOT above zero can be noticed with the data from FAVV on FFP samples. Effectively, the proportion of total positive samples is 31.9% (156 + 4 from 502), but 31.1% are below the LOT of 0.125% (w/w) and only 0.8% are above this threshold. It is to be noticed that FAVV takes two different actions towards the feed producers, depending on this classification (personal communication).

Among the official control laboratories which participated in the survey, similarities in the monitoring are noticed, but a variation in LOT is found: from zero tolerance (e.g. AGES) to 0.125% (FAVV).

It is interesting to notice that not all German laboratories apply the same LOT. In laboratory LLG, LUFA Nord-West and LUFA Rostock it is 0.2%, and in LAVES it is zero tolerance. But all Germany laboratories, as they declared in the survey, pay an attention to size and shape of the PM remnants. The problem arises that no law regulates LOT, and each country in EU is to establish its own criteria and LOT.

Sticking to a qualitative result declaration and a zero tolerance can influence the method and protocol implemented, and consequently the amount of work, as described in section 'Methods'. It is assumed, though, that for findings in very low amounts, the characteristics of the fragments (and their physical dangerousness) are evaluated by the analyst before consequences for the feed owner are driven: small fragments of paper are less harmful than pointed metal pieces. The European Regulation 767/2009 is not precise in shape and size of the fragments, which is a lack. The size and shape of PM were examined in a study by Lin et al. (2023) who conclude that it should be distinguished between PM in FFP and mixed feed according to their harm potential. It is worth to mention that U.S. Food and Drug Administration (FDA) has established guidelines for 'hard and sharp objects', defining what they deem to be a hazard, and what objects are not potentially hazardous. Canada uses the same standards for foreign objects (Payne et al. 2023). The European Union (EU) has no specific legal limits for foreign bodies in food (EU 178/2000, International Food Safety and Quality Network 2022), and neither in feed.

Most of the laboratories which participated in the survey perform analyses on PM in the frame of official control (Table 2). FAVV (BE), AGES (A), IZSPLV (I) and all six German laboratories are important actors of the control of feed in areas where FFP are largely used in animal feed. Among these nine entities, three laboratories (LUFA Nord-West, LUFA-Rostock and SGS) did not deliver data as official control entity, but analysed a high number of samples for private clients. This means that the feed producers send samples to be checked before they sell them. The results presented are not part of official control monitoring (and thus may be biased because the producers may submit mostly samples with an increased risk), but they, at least, reveal the same tendencies and show the awareness of the problem in this field. In SVA (SE), samples for private clients were also analysed, but in a lower number (23 on seven years).

At the same time, in the frame of official control in Denmark and Croatia, the number of analysed samples is low, respectively, 40 samples in six year, and 149 samples in 10 years, with no customer request. A similar situation is reported by the Canadian entity, with 84 samples analysed in the frame of monitoring in 13 years.

In Switzerland and Slovenia, from 2011 and 2013 respectively, monitoring dedicated to PM was not established, and samples were analysed in the frame of other monitoring strategies by microscopic analyses, but PM was incidentally found. It means that in parts of Europe, PM are not recognised as a major problem. Moreover, FFPs is not used for animal feeding in all EU countries in the same amount.

Conclusions

The network of feed microscopy specialists has already been involved in rapidly solving challenges of tracing problematic compounds or bodies. This is because the analysis is performed primarily by a person who can collect information and observations covering different areas and a wide range of magnification, without complex extraction protocols. The target is not only one type of molecule, and the level of precision can easily be adjusted. Consequently, the cost-effectiveness of this type of analysis is generally good.

Data sets collected were largely dispersed in terms of number of samples analysed yearly and varied slightly when considering the criteria or LOT applied for enforcement of the legislation. On the other hand, protocols used, nature of the contaminant and finally the percentage of positive samples according to the matrix (FFP or mixed feed) were not diverging much among the monitoring entities. Also, it is important to take into account not just the presence of PM but also the material, the size and shape of the fragments.

The monitoring presented here shows that over several European countries, this control is necessary because in most FFP samples, PM is present and consequently feed producers must be triggered to improve their processes allowing to separate packaging from food. Only when this is achieved can FFP be truly valorised, and safety for the animals and the food chain be guaranteed. Research is steadily going on in this field with good outcome for FFP (Mazzoleni et al. 2023).

From the entity in the Netherlands (WFSR) which established the method but did not send any data of monitoring, we know that surveillance is conducted as well on bakery by-products as on candy syrups. On the other hand, from Slovakia, we received the information that such a control is not performed. Other entities over Europe, unfortunately, neither participated in this comprehensive survey, nor gave any information about actions on the topic of PM.

Eventually, this review shows the need for monitoring, especially in countries where recycling FFP is common practice, but also in countries importing such ingredients or final products containing them.

The use of FFP reduces food waste from a circular economy perspective, as these ingredients are good for animal feed, especially for pigs, poultry and young animals. But caution is needed as it can also be a source of harmful traces of PM.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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