Analysis of mycotoxins in Spanish cow milk**.** Flores-Flores and González-Peñas. Mycotoxins in milk can have toxic effects on human and animal health. Surveillance of mycotoxin occurrence in milk is recommended. We have analyzed aflatoxins M1, B1, B2, G1 and G2, ochratoxins A and B, nivalenol, deoxynivalenol, deepoxy-deoxynivalenol, 3 and 15 acetyldeoxynivalenol, diacetoxyscirpenol, neosolaniol, fusarenon X, T-2 and HT-2 toxins, fumonisins B1, B2 and B3, sterigmatocystin and zearalenone in 191 Spanish milk samples.. Mycotoxins, extracted with acidified acetonitrile were analyzed by LC-MS/MS (triple quadrupole). None of the analyzed mycotoxins had a concentration level higher than their detection limit. Aflatoxin M1 never exceeded the level established by the European Union.

Short communication

Analysis of mycotoxins in Spanish milk

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ABSTRACT

This research paper aimed to show a survey on the presence of 22 mycotoxins in 191 Spanish cow milk samples. Mycotoxins could be carried over into animal milk and having toxic effects on human and animal health. The interaction of different mycotoxins may be additive or synergetic. Therefore, surveillance of mycotoxin co-occurrence in milk is recommended. Aflatoxins M1, B1, B2, G1 and G2, ochratoxins A and B, nivalenol, deoxynivalenol, deepoxy-deoxynivalenol, 3 and 15 acetyldeoxynivalenol, diacetoxyscirpenol, neosolaniol, fusarenon X, T-2 and HT-2 toxins, fumonisins B1, B2 and B3, sterigmatocystin and zearalenone were analyzed. Samples were treated by liquid-liquid extraction with acidified acetonitrile, followed by an acetonitrile-water phase separation using sodium acetate. The analysis was carried out by high-performance liquid chromatography coupled to a triple quadrupole mass spectrometer. None of the analyzed mycotoxins had a concentration level higher than their detection limit (0.05-10.1 µg L^{-1}). The Aflatoxin M1 in the samples never exceeded the level established by the European Union.

Key words: Milk; mycotoxin;; co-occurrence

Foods of animal origin may be contaminated with mycotoxins when they are based on or prepared with products derived from animals whose diet contained mycotoxins (Capriotti et al., 2012). Mycotoxins can appear in animal feed due to the contamination of agricultural commodities by filamentous fungi, especially those belonging to the genera *Aspergillus*, *Penicillium*, and *Fusarium* (Binder, 2007; Rodrigues and Naehrer, 2012) and therefore, the European Union has recommended limits for some of the mycotoxins and established legal limits for aflatoxins in products for animal consumption (European Commission 2006, 2013). Ruminant metabolism usually degrades mycotoxins into less toxic compounds; however, some of them can remain unaltered and they can be absorbed and accumulated in animal tissues or biological fluids, including the milk (Flores-Flores et al. 2015). Special attention has been paid to aflatoxin M1 **(AFM1**). It is formed as a degradation product in the hepatic metabolism of aflatoxin B1 (**AFB1**) in ruminants (Wu et al. 2009) and is excreted into milk. AFM1 has been classified as probably carcinogenic for humans (group 2B) (IARC, 2002): The European Community has set a maximum allowable limit of AFM1 in milk (0.05 μg kg^{-1}) (European Commission, 2010); whereas the levels of other mycotoxins in milk are not regulated. Approximately, 9.8 % of the milk samples analyzed worldwide exceeded the maximum limit set in the EU for AFM1, and it was also reported the presence of low levels of other mycotoxins in milk (Flores-Flores et al. 2015). Huang et al. (2014), detected the simultaneous presence of up to 4 mycotoxins in the analyzed milk samples: 15% were contaminated with 2 mycotoxins, 45% with 3 mycotoxins and 22% with 4 mycotoxins. This multi-exposure can change the toxic effects of mycotoxins on human and animal health due to additive, synergistic or even antagonistic phenomena (Smith et al. 2016), even when levels considered to be nontoxic of individual mycotoxins are present (Wan et al. 2013).

Therefore, the continuous surveillance of mycotoxin co-occurrence in milk is needed in order to obtain data for better risk assessment and to protect consumer and animal health. This paper shows a survey on the presence of 22 mycotoxins in 191 Spanish cow milk samples.

Methanol (LC-MS grade), formic acid (mass spectrometry grade, purity > 98%), ammonium formate (analytical grade) and sodium acetate (anhydrous, HPLC grade > 99.0%) were purchased from Sigma-Aldrich (USA) and acetonitrile (HPLC grade) from Merck (Germany). Deionized water (> 18 MΩ/cm resistivity) was purified in an Ultramatic Type I system from Wasserlab (Spain). All mycotoxins (purity ≥ 98%) were obtained from Sigma-Aldrich (USA) in solution except for ochratoxin A which was purchased in powder form.

Three mixed stock solutions (1, 2 and 3) were prepared by dilution of appropriate volumes of each mycotoxin standard solution in 10 mL of acetonitrile as previously described (Flores-Flores and González-Peñas 2015, 2017). Mixed stock solution 1 contained nivalenol (**NIV**), deoxynivalenol (**DON**), deepoxy-deoxynivalenol (**DOM-1**), fusarenon X (**FUS-X**), neosolaniol (**NEO**), 3-acetyldeoxynivalenol (**3-ADON**), 15-acetyldeoxynivalenol (**15-ADON**), diacetoxyscirpenol (**DAS**), HT-2 toxin (**HT-2**) and T-2 toxin (**T-2**). Mixed stock solution 2 contained AFB1, aflatoxin B2 (**AFB2**), aflatoxin G1 (**AFG1**), aflatoxin G2 (**AFG2**), AFM1, ochratoxin A (**OTA**), ochratoxin B (**OTB**), zearalenone (**ZEA**) and sterigmatocystin (**STC**). Mixed stock solution 3 contained fumonisin B1 (**FB1**), fumonisin B2 (**FB2**) and fumonisin B3 (**FB3**). Mixed stock solutions 1 and 2 were stored at -20 ºC. Mixed stock solution 3 was prepared and used daily due to the instability of fumonisins in acetonitrile. For a better understanding of this paper, the mycotoxins contained in mixed stock solution 1 will be referred to as mycotoxin group 1 and mycotoxins from mixed stock solutions 2 and 3 will be referred to as mycotoxin group 2. Due to the toxicity of these compounds, all of them were handled in solution using gloves and a face shield. In addition, low-light conditions were established during handling so as to prevent photo-instability.

One hundred and seven full cream milk samples were purchased from supermarkets in Spain between September 2013 and April 2016 due to the fact that Spanish families purchase more than 50% of the liquid milk consumed in this type of establishment (MAGRAMA 2015, 2016). Between 2010-2014 (INE, 2016), added together, Galicia, Castile and Leon, Andalusia, Catalonia, Asturias, Cantabria, Navarra, Castile-La Mancha and Basque Country make up 93% of the total production of cow milk in the country in 2014. Samples were from 26 collection centers located in the high milk production regions (figure 1). Samples were opened and analyzed during 1-2 days.

Also, eighty-four raw milk samples were collected in March 2016. All of them were from dairy farms located either in Navarra, La Rioja, Basque Country or Catalonia. One of the raw samples was taken from a cow with signs of disease of unknown origin. None of these samples suffered any treatment procedure after collection, with the exception of the addition of azidiol (sodium azide/chloramphenicol) a preservative compound frequently used by milk testing laboratories in Spain (Llopis et al. 2013) as a preservative. Samples were analyzed within the week that they were collected and maintained at 4 ºC until analysis. Prior to chromatographic analysis, milk samples were treated following the procedures previously developed by our group (Flores-Flores and González-Peñas 2015, 2017). Briefly, 1 mL of milk was poured into a tube for the analysis of mycotoxin group 1 and another 1 mL was poured into a second tube for the analysis of mycotoxin group 2. Each tube was extracted with acidified acetonitrile. After centrifugation, the upper phase of each tube was transferred to another clean tube and water-acetonitrile phases separation was induced by the addition of sodium acetate. Next, each acetonitrile phase was dried and the residue from each one of the tubes was reconstituted with LC mobile phase. In addition, the two groups of mycotoxins were analyzed in separate runs with different separation conditions, as explained below.

An Agilent Technologies (Germany) 1200 LC system was used. The chromatographic column was an Ascentis Express C18, 2.7 µm particle size, 150 mm x 2.1 mm from Supelco Analytical (USA) maintained at 45 ºC. The mobile phase consisted of solution A (5 mM ammonium formate and 0.1% formic acid in water) and solution B (5 mM ammonium formate and 0.1% formic acid in methanol:H_2O 95:5) in gradient conditions. Fifteen µL and 20 µL were injected for mycotoxin group 1 and 2, respectively. Flow rate was 0.4 mL min^{-1}. Detection was carried out using a 6410 Triple Quad LC-MS/MS System from Agilent Technologies (Germany) equipped with an electrospray ionization interface. MS operation conditions were: capillary voltage at 4000 V, drying gas was high purity nitrogen at 350 ºC, 9 L/min and 40 psi. In addition, ultra-high purity nitrogen (99.999%, Praxair, Spain) was used inside the collision cell. Selected reaction monitoring was used for data collection. MS parameters for identifying each one of the mycotoxins were those previously reported by our group (Flores-Flores and González-Peñas 2015, 2017).

Validation of the two developed methodologies has been previously described (Flores-Flores and González-Peñas 2015, 2017). Limits of detection (**LOD**) and quantification (**LOQ**), linearity, precision, accuracy, recovery, matrix effect, and stability were studied for both methods. Recovery values were between 53.8 and 94.4% for all the mycotoxins, except for fumonisin B1 which was 42.1%. RSD (%) values (in intermediate precision conditions) were lower than 15% for all the mycotoxins. Matrix effect appeared for all of the mycotoxins, and matrix-calibration curves were constructed for all of them. Detection limits were between 0.02 and 10.14 µg L^{-1} for all the mycotoxins (table 1). For AFM1, the only mycotoxin for which a maximum limit of 0.05 µg kg^{-1} has been established in the UE, a LOD of 0.025 µg L^{-1} was achieved.

All the samples were analyzed as analytical sequences, including quality control samples (two in every ten) prepared by spiking milk at the LOQ and the highest levels in the quantification range of each one of the mycotoxins. In the case of mycotoxin presence in the sample, the sample was re-analyzed along with calibration samples prepared by spiking milk samples, using the same procedure as when validation studies were performed.

Raw milk samples contained azidiol, and therefore, the effect of the presence of this compound in the response of mycotoxins in the detector was evaluated. Milk containing this product was fortified at LOQ and the highest level in the quantification range of each mycotoxin. The responses (peak areas) obtained were compared with those obtained for mycotoxins in fortified commercial milk samples at the same concentration levels. The experiment was carried out on three consecutive days. The relationship between the mean areas in both types of samples at each one of the concentration levels (LOQ and the highest level in each range) was less than 15% for all the mycotoxins. Thus, we considered no additional matrix effects due to the azidiol. However, when analyzing raw milk samples, we prepared the quality control samples using raw milk containing azidiol as matrix. Figure 2 shows examples of the chromatograms obtained for both groups of mycotoxins in raw milk samples.

We did not find levels of mycotoxins higher than the respective LOD values in any of the analyzed samples. Therefore, and with respect to AFM1, the only mycotoxin for which a permissible maximum limit has been established in milk within the European Union, all tested samples complied with legislation in terms of this mycotoxin.

Aguilera-Ruiz et al. (2011) analyzed 8 mycotoxins in 15 milk samples purchased in the province of Almeria. Beltrán et al. (2013) analyzed 18 mycotoxins in different food matrices, including 10 milk samples from the region of Valencia, and Beltrán et al. (2011) analyzed 6 mycotoxins in 2 raw milk samples from the city of Castellon. None of these studies found mycotoxins levels. The LOQs from these studies were higher than the LOQs of the methods used in this study. In addition, González-Osnaya et al. (2008) analyzed OTA in 16 samples of whole milk purchased from the province of Valencia; none of them contained OTA above the detection level of the method.

In a previous publication, we carried out a review regarding the presence of AFM1 levels in milk collected in Europe (Flores-Flores et al. 2015). From 13566 analyzed milk samples, only 119 (0.9%) had AFM1 in levels higher than the maximum permitted by the EU. From a geographical point of view, 117 out of 119 positive samples for AFM1 correspond to countries in the Adriatic Sea region. In Spain, Rodríguez et al. (2003) and Cano-Sancho et al. (2010) did not detect AFM1 levels higher than those established by EU legislation when analyzed milk samples from Leon and Catalonia, respectively. Thus, the results obtained in this study (none of the analyzed mycotoxins had a concentration level higher than their detection limit and aflatoxin M1 never exceeded the level established by the European Union). coincide with those found in the literature and they are encouraging and demonstrate a low risk of mycotoxin contamination in Spanish milk. However, due to the importance of milk due to its economic impact as well as its elevated consumption, especially by a vulnerable group such as children, we recommend the carry-over of periodic surveillance programs.

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Table

Table 1. Linear range, LOQ (lowest level of the range) and LOD of the validated methods (Flores-Flores and González-Peñas 2015, 2017)

	Range (µg L^{-1})	LOD (µg L^{-1})		Range (µg L^{-1})	LOD (µg L^{-1})		Range (µg L^{-1})	LOD (µg L^{-1})
NIV	20.2 - 202.3	10.1	AFG2	0.15-1.50	0.075	FB2	2.50-25.00	2.500
DON	5.0 - 50.3	2.5	AFM1	0.05-0.50	0.025	FB3	2.50-25.00	0.625
DOM-1	3.0 - 30.3	1.5	AFG1	0.10- 1.02	0.025			
FUS-X	3.7 - 37.0	1.9	AFB2	0.04-0.40	0.020			
NEO	0.2 - 2.0	0.1	AFB1	0.04-0.40	0.020			
3-ADON	1.0 - 10.0	0.5	OTB	0.05-0.50	0.050			
15-ADON	2.0 - 20.2	1.0	FB1	10.14-50.70	10.140			
DAS	0.16 - 1.6	0.08	ZEA	0.51-5.09	0.510			
HT-2	0.8 - 8.0	0.4	STC	0.50-5.02	0.125			
T-2	0.1 - 1.0	0.05	OTA	0.20-1.00	0.200			

Limit of Quantification (LOQ), Limit of Detection (LOD), nivalenol (NIV), deoxynivalenol (DON), deepoxy-deoxynivalenol (DOM-1), fusarenon X **(**FUS-X), neosolaniol (NEO), 3-acetyldeoxynivalenol (3-ADON), 15-acetyldeoxynivalenol (15-ADON), diacetoxyscirpenol (DAS), HT-2 (HT-2), T-2 (T-2), aflatoxin G2 (AFG2), aflatoxin M1 (AFM1), aflatoxin G1 (AFG1), aflatoxin B2 (AFB2), aflatoxin B1 (AFB1), ochratoxin B (OTB), fumonisin B1 (FB1), zearalenone (ZEA), sterigmatocystin (STC), ochratoxin A (OTA), fumonisin B2 (FB2) and fumonisin B3 (FB3).

Figure captions

Figure 1. Distribution of analyzed commercial milk samples classified by collection center code

Figure 2. Superposed quantification products ions obtained after analysis of mycotoxin group 1 (A and B) and 2 (C and D). (A and C) Raw milk sample (containing azidiol) fortified at Limit of Quantification and (B and D) non-fortified raw milk sample (containing azidiol). Group 1: (NIV), deoxynivalenol (DON), deepoxy-deoxynivalenol (DOM-1), fusarenon X **(**FUS-X), neosolaniol (NEO), 3-acetyldeoxynivalenol (3-ADON), 15-acetyldeoxynivalenol (15-ADON), diacetoxyscirpenol (DAS), HT-2 (HT-2), T-2 (T-2). Group 2: aflatoxin G2 (AFG2), aflatoxin M1 (AFM1), aflatoxin G1 (AFG1), aflatoxin B2 (AFB2), aflatoxin B1 (AFB1), ochratoxin B (OTB), fumonisin B1 (FB1), zearalenone (ZEA), sterigmatocystin (STC), ochratoxin A (OTA), fumonisin B2 (FB2) and fumonisin B3 (FB3).

Flores-Flores. Figure 1.

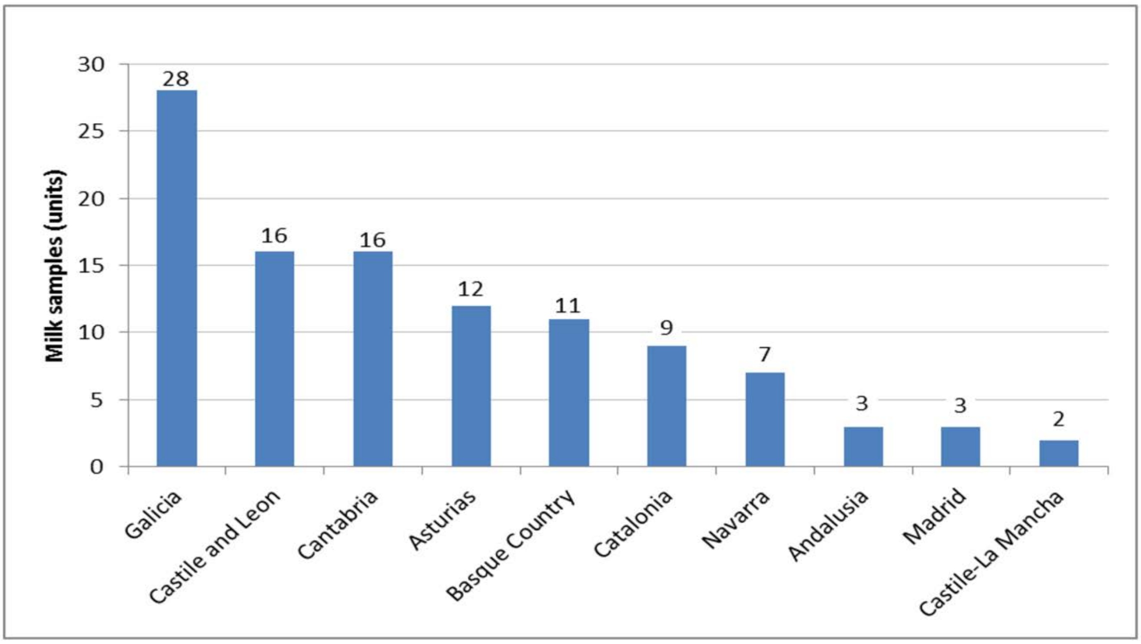
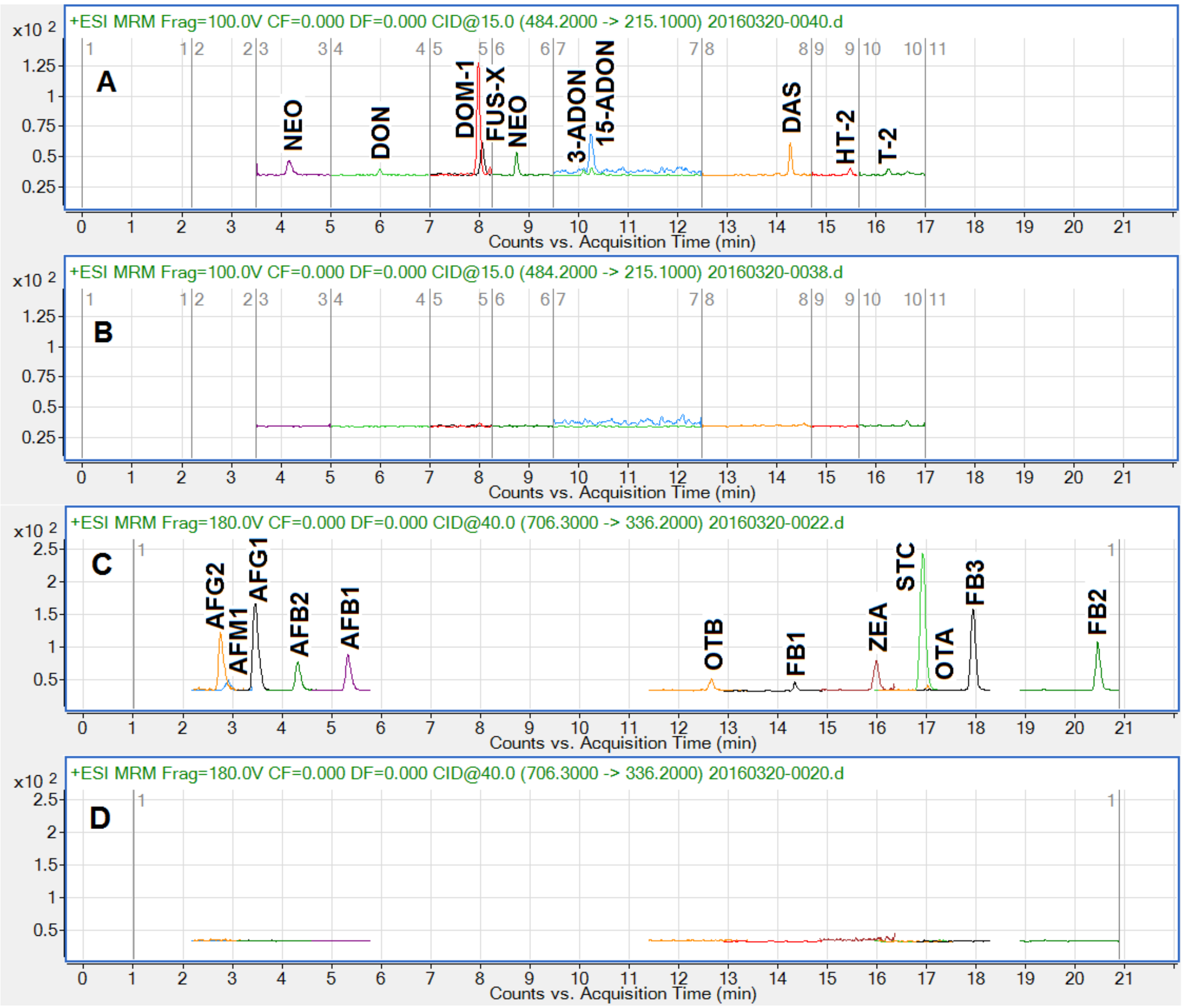


Figure 2.


+ESI MRM Frag=100.0V CF=0.000 DF=0.000 CID@15.0 (484.2000 -> 215.1000) 20160320-0040.d
A
NEO
DON
DOM-1
FUS-X
NEO
3-ADON
15-ADON
DAS
HT-2
T-2
Counts vs. Acquisition Time (min)
+ESI MRM Frag=100.0V CF=0.000 DF=0.000 CID@15.0 (484.2000 -> 215.1000) 20160320-0038.d
B
Counts vs. Acquisition Time (min)
+ESI MRM Frag=180.0V CF=0.000 DF=0.000 CID@40.0 (706.3000 -> 336.2000) 20160320-0022.d
C
AFG2
AFM1
AFG1
AFB2
AFB1
OTB
FB1
ZEA
STC
OTA
FB3
FB2
Counts vs. Acquisition Time (min)
+ESI MRM Frag=180.0V CF=0.000 DF=0.000 CID@40.0 (706.3000 -> 336.2000) 20160320-0020.d
D
Counts vs. Acquisition Time (min)