Montevideo, 30 de abril de 2019.

Sr. Ariel Martínez

Presente

A continuación se detallan los principales avances en la consultoría: Ajuste de tecnología de análisis rápido de glifosato, del proyecto “Fortalecimiento de la capacidad exportadora del sector apícola del Uruguay a través del aseguramiento de la inocuidad y la incorporación de tecnología” financiado por ANDE.

Sin otro particular

Saluda atentamente

Dr. Eduardo Boido

1- Desarrollo de un método rápido de medida a través de NIR

Se está desarrollando un método de medida rápido de glifosato en miel utilizando la tecnología NIR.

Con este fin se utilizó como instrumento de medida un NIR de la marca Büchi modelo NIRFlex N-500 con la celda de medida para sólidos NIRFlex perteneciente al Departamento de Proyectos Forestal del LATU.

Las muestras utilizadas para el desarrollo de la calibración y validación del modelo fueron 5 muestras de miel que se contaminaron en distintos niveles de glifosato, como se muestra en la Tabla 1. Por otra parte se utilizaron otro grupo de 5 muestras para la predicción de la concentración de glifosato a partir de la calibración obtenida. Las 5 muestras originales y las 5 muestras posteriormente utilizadas en la predicción fueron analizadas por HPLC-MS/MS en el Istituto San Michele all'adige.

Tabla 1. Muestras utilizadas para el desarrollo de la calibración y la validación del método de medición de glifosato por NIR.

Muestra	Concentración	Muestra	Concentración	Muestra	Concentración	Muestra	Concentración
1	0,0	16	78,6	36	113,8	53	70,2
2	0,0	17	78,3	37	113,7	54	113,7
3	0,0	18	78,3	40	94,0	55	113,3
4	9,8	19	113,0	41	94,0	60	0,0
5	9,8	20	113,5	42	103,8	61	0,0
6	9,7	21	112,8	43	103,8	62	51,0
7	26,0	22	163,9	44	129,2	63	51,0
8	25,4	23	166,8	45	129,2	64	113,6
9	25,5	24	161,5	46	172,3	65	113,9
10	35,4	30	0,0	47	172,8	70	0,0
11	35,2	31	0,0	48	258,8	71	0,0
12	35,3	32	25,3	49	258,3	72	25,5
13	51,0	33	25,6	50	35,0	73	25,5
14	51,0	34	51,1	51	35,0	74	78,7
15	51,2	35	50,9	52	70,3	75	78,5

Con el fin de obtener el mejor modelo de calibración, se ensayaron diferentes pretratamientos de datos y se utilizó un set de muestras para la validación, obteniéndose los mejores parámetros en la calibración Project 3 (Figura 1). La calibración Project 3 se realizó con un suavizado de datos mediante promedio de 3 puntos y posterior derivada segunda del espectro mediante método de Savitzky-Golay.

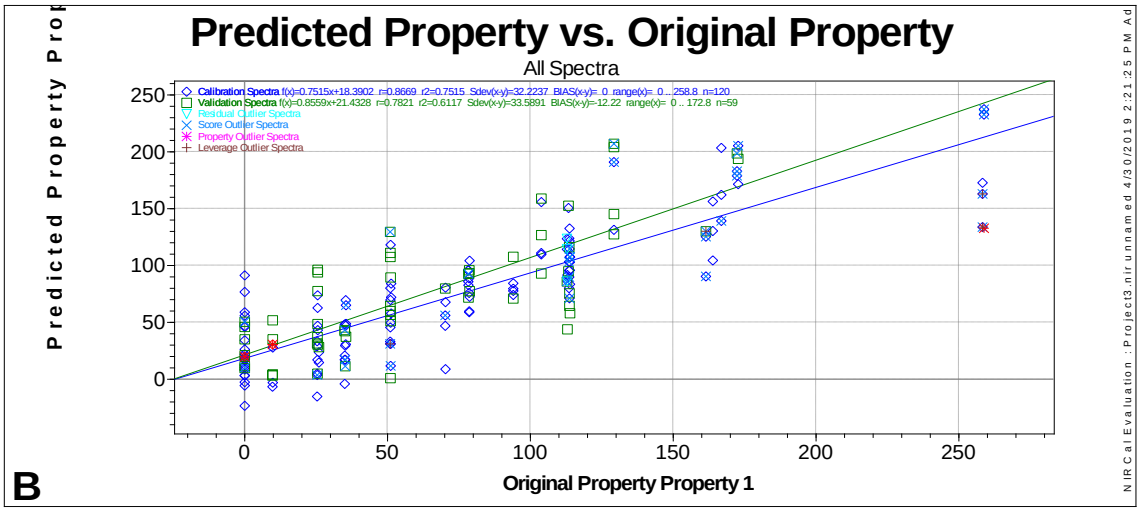

Figura 1. Valores predecidos de glifosato en muestras de miel vs concentración real de glifosato aplicando la calibración obtenida en Project 3.

En la Tabla 2 se muestra la predicción de los valores obtenidos con la calibración Project 3 y el valor real obtenido por HPLC-MS/MS.

Tabla 2. Valores reales y predecidos del batch de pedicción

PREDECIDO	PROMEDIO	REAL
116	114	48
116		
111		
88	76	74
92		
48		
130	107	110
76		
115		
95	116	192
136		
117		
85	69	123
97		
24		

Según los datos presentados en la Tabla 2, solo una de las muestras que presentó un valor real de 48 ppb fue erróneamente clasificada como superando el límite establecido para exportación en la unión europea.

Cabe destacar que la calibración fue realizada utilizando 5 mieles que se contaminan a niveles distintos y la predicción se realizó con mieles diferentes a las utilizadas en el desarrollo del modelo.

Se realizó otra prueba en la cual el modelo de calibración y validación se realizó con 10 mieles (las 5 iniciales y las 5 que se utilizaron para la predicción) y para predecir se sacaron 5 muestras contaminadas de las que en la experiencia anterior se habían usado para la calibración. Se

obtuvo la calibración que se muestra en la Figura 2 y los resultados de la predicción se muestran en la Tabla 3.

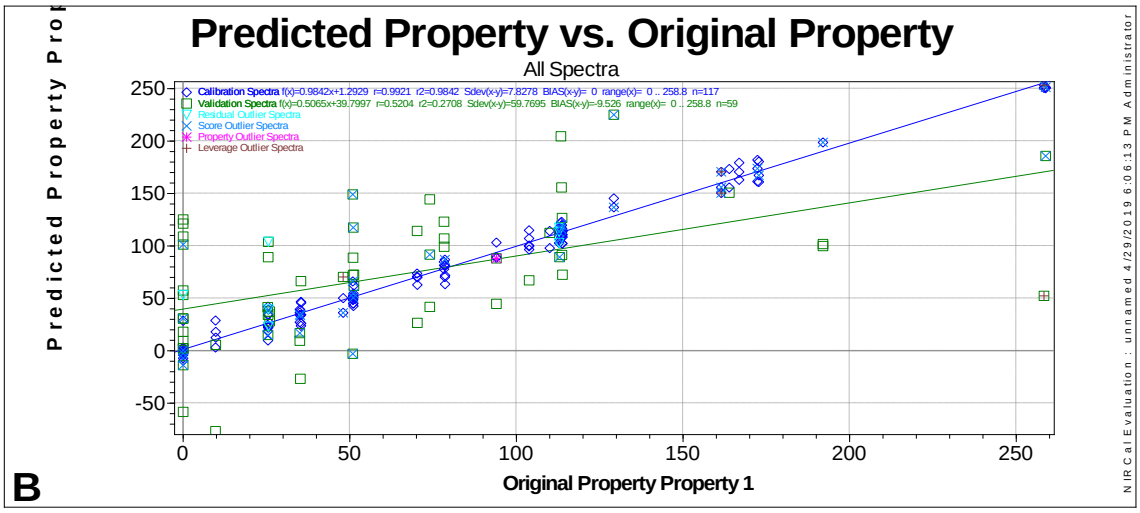

Figura 2. Valores predecidos de glifosato en muestras de miel vs concentración real de glifosato utilizando la nueva calibración.

Tabla 3. Valores predecidos del nuevo batch de predicción

PREDECIDO	PROMEDIO	REAL
13	36	25
25		
69		
55	85	79
99		
102		
181	125	129
94		
100		
50	63	51
101		
39		
39	33	10
10		
52		

Según los datos presentados en la Tabla 3, todas las muestras son bien clasificadas con mayor o menor contenido de glifosato según el límite establecido para exportación en la unión europea.

Dadas las calibraciones obtenidos hasta el momento (Project 3) y con la mejora en la predicción obtenida en la segunda calibración realizada (al variar el batch de predicción), se concluye que es necesario incluir mayor número de muestras reales al modelo, ya que cuanto mayor variabilidad en la matriz (miel) se incluya en el modelo se nota una mejora en los valores que es capaz de predecir el mismo.

Búsqueda bibliográfica de métodos reportados para el análisis de Glifosato en miel y otras matrices.

Se realizaron búsquedas bibliográficas en los dos principales bases de datos de literatura científica disponibles: SCOPUS y SCIFINDER-CAS.

En la Tabla 4 se muestran los resultados obtenidos para determinación de glifosato en muestras de miel que se encuentran reportados en la bibliografía.

Tabla 4. Métodos reportados para glifosato en miel

Referencia	Metodología	LOQ
L. Pareja et al. Evaluation of glyphosate and AMPA in honey by water extraction followed by ion chromatography mass spectrometry. A pilot monitoring study. Analytical Methods 2019, 11, 2123-2128	Cromatografía iónica y espectrometría de masa de alta resolución (IC-HRMS Q-Orbitrap)	Glifosato: 5 µg/kg Rango lineal 5-500 µg/kg AMPA: 20 µg/kg
L.M. Chiesa et al. Detection of glyphosate and its metabolites in food of animal origin based on ion-chromatography-high resolution mass spectrometry (IC-HRMS)	Cromatografía iónica y espectrometría de masa de alta resolución (IC-HRMS orbitrap analyses)	4.30-9.26 µg/kg
T.S.Thompson et al. Determination of glyphosate, AMPA, and glufosinate in honey by online solidphase extraction-liquid chromatography-tandem mass spectrometry. Food Additives & Contaminants, Part A 2019, 36, 434-446	Cromatografía líquida y espectrometría de masa (HPLC-MS/MS)	1 µg/kg
C.J. Berg et al. Glyphosate residue concentrations in honey attributed through geospatial analysis to proximity of large-scale agriculture and transfer off-site by bees. PLoS One 2018, 13, e0198876/1-18	Abraxis' ELISA	15 µg/L (15 ppb)
O. Zoller et al. Glyphosate residues in Swiss market foods: monitoring and risk evaluation. Food Additives & Contaminants, Part B: Surveillance 2018, 11, 83-91	Cromatografía líquida y espectrometría de masa (HPLC-MS/MS)	0.5-2.5 µg/kg
B. Li et al. Determination of Glyphosate and Aminomethylphosphonic Acid Residues in Foods Using High Performance Liquid Chromatography-Mass Spectrometry/Mass Spectrometry. Chinese Journal of Chromatography 2007, 25, 486-490	Cromatografía líquida y espectrometría de masa (HPLC-MS/MS)	50 µg/kg

De los datos reportados en bibliografía las metodologías utilizadas para cuantificar glifosato en miel son ELISA y HPLC-MS/MS.

De las bases de datos utilizadas se seleccionaron 130 documentos donde se determina glifosato en otras matrices por distintos métodos analíticos. En este momento se están purificando estos datos con el fin de evaluar su posible utilización en miel.

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