8º CONGRESO LATINOAMERICANO SOBRE RESIDUOS DE PLAGUICIDAS

Pesticides residues reduction from fish during household cooking

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ABSTRACT

Pesticide residues are distributed in the environment and become pollutants for biotic and abiotic systems, threatening their stability and representing a public health hazard[1,2,3]. Fish are exposed to contaminants through direct contact with water or sediment, and by of other already contaminated organisms' intake. The objectives of this work were an analytical methodology validation for the simultaneous organochlorine pesticides determination in fish muscle, and the study of the cooking effect on their initial content. The analytes extraction was carried out by QuEChERS (Quick Easy Cheap Effective Rugged Safe) methodology, and they were determined by gas chromatography with a micro electron capture detector (GC-µECD). The results were confirmed by a gas chromatograph coupled with a mass spectrometer (GC-MS). The methodology validation was carried out following the SANTE Guide recommendations[4]. The matrix added calibration curve was linear in the range of 0.001 to 0.5 mg/l, with regression coefficients R^2 greater than 0.99. Precision was evaluated at three levels, 0.001; 0.05 and 0.5 mg/l, with a relative standard deviation (RSD) lower than 10 % (n=5). Recovery was studied at the same concentrations (n=3), with values between 88 and 110%.

The expanded uncertainty estimation was performed, being less than 20% in all cases. Then, the household cooking effect on the initial pesticides concentration in fish was evaluated. The study was performed with an electric oven, in triplicate. A thermocouple was used in order to verify that the temperature at the thermal center of the samples were 71 °C. After cooking the fish samples, the concentration of each compound was determined. The thermal destruction percentage during the cooking process was 55.5; 50.4; 44.0; 37.6; 32.6; 52.2; 44.1 and 63.7% for transchlordane, endrin, endosulfan, dieldrin, heptachlor epoxy A; cischlordane; lindane and heptachlor epoxide B, respectively. No reduction was observed for aldrin and DDD. The statistical analysis of the results indicated that the method is linear, and has high sensitivity, accuracy and precision, being useful for the determination of organochlorine compounds in fish muscle. It can be concluded that cooking at 71 °C in the thermal center ensures the partial elimination of the analytes studied, with the exception of aldrin and DDD. It is considered relevant to continue the research in order to evaluate the effect that other cooking conditions will have, allowing optimization of the results.

METODOLOGÍA

❖ **TRATAMIENTO PREVIO DE LAS MUETRAS**

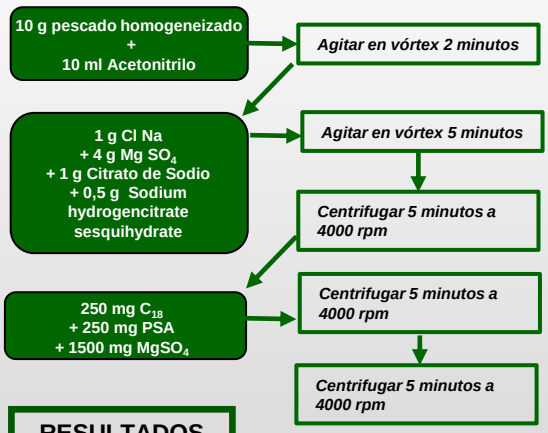
Homogeneización total de las muestras de musculo de pescado, utilizando una procesadora semi-industrial de 3000rpm durante 25 minutos. Almacenamiento a -18ºC hasta su posterior análisis. Las determinaciones analíticas se efectuaron por quintuplicado (n=5 y α=0.05)

❖ **ESTANDARES, BLANCO DE MUESTRAS Y MUESTRAS ADICIONADAS**

- ***Soluciones estándares:*** transclordano, endrin, endosulfán, dieldrin, heptacloro epoxi A, heptacloro epóxido B, cisclordano, lindano, aldrin y DDD.
- Muestras adicionadas con 0,001 a 0,5 mg/l.

❖ **PROCESO DE COCCIÓN:** 71°C en el centro térmico.

❖ **PROCESO EXTRACTIVO: QuEChERS**



❖ **CONDICIONES CROMATOGRAFICAS**

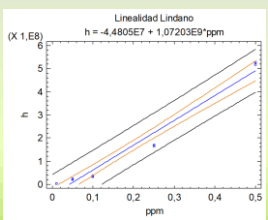
Cromatógrafo GC 6890N- 2

Equipo	GC - µECD
Columna	HP-5MS (30 m × 0,25 mm i.d., 0,25 µm)
Rampa Horno	80ºC (0,2 min), rampa 10ºC/min hasta 280°C (3 min), rampa 15ºC/min hasta 290°C (1 min)
Otras Condiciones	Temp. inyector 250°C; Temp. detector 330°C; Volumen injección: 1 µL Gas carrier: N_2 Flujo: 1ml/min

RESULTADOS

El análisis estadístico de los resultados indicó que el método es lineal, y presenta alta sensibilidad, exactitud y precisión, siendo útil para la determinación de compuestos organoclorados en músculo de pescado

Rango (ppm)	0,001 - 0,5
R^2	> 0,99
Precisión RSD (n=5)	< 10%
Recuperación (n=3)	88 - 110%
Incertidumbre expandida	< 20%



Porcentaje de destrucción térmica durante el proceso de cocción (n=3)	
Transclordano	55,5
Endrin	50,4
Endosulfan	44,0
Dieldrin	37,6
Heptacloro epoxi A	32,6
Cisclordano	52,2
Lindano	44,1
Heptacloro epoxi B	63,7

CONCLUSIONES

Se puede concluir que la cocción a 71°C en el centro térmico asegura la eliminación parcial de los analitos estudiados, a excepción de aldrin y DDD. Se considera relevante continuar la investigación a fin de evaluar el efecto que tendrán otras condiciones diferentes de cocción permitiendo optimizar los resultados