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Multiclass Multipesticide Residue Analysis in Hake on the Market in Serbia

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The aim of this study was an assessment of pesticide residues in hake (*Merluccius merluccius*) on the Serbian market.

The fish samples were comminuted, extracted and cleaned by a modified QuEChERS method. The prepared samples were analysed by gas chromatography – mass spectrometry (GC/MS) technique in the selected ion monitoring mode (SIM) using one target and three qualifier ions for each analyte. 59 pesticide compounds were analyzed in the samples using the Oregon pesticide standard mixtures. The identified compounds were quantified against the authentic standards. More than 100 samples were analysed in this study.

The results confirmed presence of carbaryl (in concentration range from not detectable (ND) to 7.4 µg kg⁻¹), cyfluthrin (sum of isomers; from ND to 107.5 µg kg⁻¹), cypermethrins (from ND to 14.3 µg kg⁻¹), metalaxyl (from ND to 8.6 µg kg⁻¹), diazinon (from ND to 11.2 µg kg⁻¹), bifenthrin (from ND to 11.6 µg kg⁻¹), imazalil (from ND to 15.0 µg kg⁻¹), methyl parathion (from ND to 18.2 µg kg⁻¹), *p*,*p*'-DDT (from ND to 40.7 µg kg⁻¹), chlorpyriphos (from ND to 12.7 µg kg⁻¹), and endosulfan (from ND to 19.7 µg kg⁻¹) in nine fish samples.

Cyfluthrin and p,p'-DDT had the highest frequencies of occurrence and were detected in 4 samples. Cypermethrins, imazalil, methyl parathion, and endosulfan were found in two samples each while carbaryl, cypermethrins, metalaxyl, diazinon, bifenthrin, and chlorpyriphos were found in one of the samples each.

Except from carbaryl and metalaxyl, all other pesticides quantified in this study were found in concentrations higher than a maximum residue level (MRL) of 0.01 mg/kg as set by EU legislation on MRLs [1]. Considering the fact that most of these pesticides are not on the EU pesticide Watch List, these results point to the need for expansion of this list.

p,p'-DDT belongs to a group of organochlorine pesticides - the group of compounds that are highly toxic to mammals, resistant to degradation and prone to bioaccumulation [2]. Because of that, its presence in food is usually a cause for increased concern. However, the p,p'-DDT concentrations measured in this study are lower than 5 ppm which is the action limit (the threshold at or above which the product should be removed from the market) set by FDA [3] and because of that it is not expected to pose any health risk to the humans consuming these products.

According to the results of the quality testing of hake on the market in Serbia, it can be concluded that these analyses were proven justified and that they should be continued in the future, as well as the control of the application of all pesticides that were found in this study in concentrations higher than the MRL.

Acknowledgements

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References

[1] Regulation (EC) No 396/2005 of the European Parliament and of the Council of 23 February 2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin and amending Council Directive 91/414/EE

[2] R. Jayaraj, P. Megha, P. Sreedev, *Interdisciplinary toxicology* (2016) 90-100.

[3] Food and Drug Administration (FDA), Environmental chemical Contaminant and pesticide tolerances, action levels and guidance levels for fish and fishery products (Compliance Policy Guide Sec.575.100. Pesticide Chemical Residues in Food – Enforcement Criteria, CPG 7141.01), 2001.