DETERMINATION OF QUINOLONES IN FISH MUSCLE PLUS SKIN BY ULTRA HIGH - PERFORMANCE LIQUID CHROMATOGRAPHY WITH PHOTO – DIODE ARRAY DETECTION

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Quinolones are antimicrobial drugs widely used in fish farming for prophylactic and therapeutic purposes. During the last decades, they have received growing attention for their potential in fish therapy against various systemic bacterial infections. However, the potential hazards associated with their residues in the edible tissues of farmed fish include allergies, toxic effects and antimicrobial resistance. For this reason European Union has established Maximum Residue Limits (MRLs) in order to limit human exposure to these drugs. Oxolinic acid and flumequine are members of the older generation of quinolone drugs which are less efficient but still regularly used in fish farming. Enrofloxacin, ciprofloxacin, danofloxacin and sarafloxacin are later – generation fluoroquinolones. Nalidixic acid is one of the earliest – known members of the quinolone class with limited activity.

In this study a Ultra High Performance Liquid Chromatography and Photo Diode Array (UHPLC - PDA) method was developed and validated for the determination of the aforementioned quinolones in fish muscle plus skin tissues using enoxacin as internal standard. The analytes were extracted by acetonitrile followed by the salting out of water from the sample using anhydrous magnesium sulfate, sodium chloride and buffering citrate salts to induce liquid-liquid partitioning. Fish tissue extracts were subjected to dispersive solid phase extraction (dSPE) using a C18 sorbent and were then ready for analysis. Chromatographic analyses were performed using the UHPLC system model Acquity (Waters, USA) and seperations were achieved on a BEH C18 2.1×100 mm, 1.7 µm analytical column kept at 50 °C. Mobile phase was consisted of water and acetonitrile containing formic acid 0.1 % (v/v) and was pumped at a flow rate of 0.4 mL min⁻¹ using a gradient elution programme. Quinolone detection was achieved by a PDA detector monitored at 260, 275 and 280 nm and by using the Empower software (Waters, USA). The developed method was validated for fish muscle plus skin tissue in compliance with the requirements set by Commission Decision 2002/657/EC.

KEYWORDS: Quinolones, Fish muscle plus skin, dSPE, UHPLC, Decision 2002/657/EC

REFERENCES: