Residues of endosulfan in carp as an indicator of exposure conditions

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Endosulfan is an organochlorine insecticide, consisting of a mixture of two isomers: alpha and beta endosulfan. Endosulfan residues were determined in livers and gills of carp exposed to lethal and sublethal concentrations of endosulfan. The fish which were exposed to a lethal concentration contained the highest residue level in both liver and gills. In carp liver, the percentage of beta endosulfan in the residue decreased with time between exposure and collection of samples whereas the percentage of endosulfan sulphate increased. Carp killed by exposure to endosulfan had a significantly greater ratio of beta to alpha endosulfan and a significantly greater percentage of beta endosulfan in their livers. There was no such clear relationship for the residue composition in fish gills. The determination of residue composition, in particular the percentage of beta endosulfan or the ratio of beta to alpha isomers is recommended in investigations of fish kills when endosulfan is a suspected cause.

Keywords: organochlorines; fish kill; carp; endosulfan.

Introduction

Endosulfan is an organochlorine insecticide which is less persistent in the environment but more toxic to fish than other organochlorines (for a review see Goebel et al. (1982)). Technical endosulfan consists of two isomers: alpha and beta endosulfan, which are mixed together in a ratio of approximately 7:3. Fish metabolize endosulfan to endosulfan sulphate, which is as toxic as the parent compound and then to relatively non-toxic metabolites; endosulfan diol, endosulfan ether and endosulfan hydroxyether.

Residues of organochlorines in fish have often been used as indicators of contamination of aquatic environments (Phillips 1980). Residue level is dependent on the concentration to which the fish were exposed, exposure time and time between exposure and sample collection. In addition, the results can be confounded by other factors such as species of fish and the sex, size and lipid content of individual animals.

Endosulfan residues were detected in wild fish from the areas where endosulfan is used (Pick et al. 1981, Matthiessen et al. 1982, Nowak 1990, Nowak and Julli 1991). The residues usually consisted of the alpha isomer and endosulfan sulphate, which indicates lower persistence of the beta than the alpha isomer in fish (Nowak and Julli 1991). Catfish, Tandanus tandanus, experimentally exposed to endosulfan under laboratory conditions contained the highest residue levels in liver, gills, kidney and brain (Nowak 1992). Only those fish which were exposed to endosulfan for a short time...
before they were sacrificed contained residues of beta endosulfan in their livers. This suggests that the composition of endosulfan residues could relate to exposure conditions and that a greater relative proportion of beta endosulfan indicates a more recent exposure.

Due to its high toxicity to fish, endosulfan is often suspected as the cause of fish kills in cotton-growing regions of New South Wales and Queensland and other areas where it is used. Since it is not very persistent in water, analyses of water samples rarely provide sufficient cause and effect evidence. Residues in dead fish would seem to be better indicators of exposure. Unfortunately, problems arise if the residue level is within the range usually found in live fish or if there is no available background information on residue levels in fish from a particular area. However, the presence of beta endosulfan in the tissue could be used to estimate exposure conditions, provided that the field observations were confirmed by a controlled experiment.

Materials and methods

Adult common carp, *Cyprinus carpio*, were collected by overnight gill netting. Fish of both sexes were used; the mean weight of the fish was 199 g (SE = 22.14). Before the experiment started, fish were acclimated to laboratory conditions for at least 1 week. The acclimation and experimental conditions were as follows: mean temperature 24.5 °C (SE = 0.14), mean pH 7.0 (SE = 0.05) and mean oxygen saturation 81% (SE = 0.80).

Four fish were each assigned to 210 l tanks using random selection procedures and two tanks were randomly assigned to each treatment. There was no statistically significant difference in the size of the fish (ANOVA, *p* = 0.1812) or lipid content of the gills or livers (ANOVA, *p* = 0.4734) of the fish assigned to different treatments. Three treatments were used in the experiment: one lethal and two sublethal exposures. In the former treatment the fish were exposed to a lethal concentration of endosulfan (1 ppm) and collected when moribund. In the sublethal treatments the fish were exposed once to 0.001 p.p.m. of endosulfan and collected after either 1 or 14 days post-exposure. Endosulfan was applied as endosulfan 350 g l⁻¹ EC (Thiodan).

Fish were sacrificed at the end of the treatment period, dissected and their livers and gills collected for endosulfan analysis. To ensure independence of results, livers and gills were obtained from different individuals. Thus, two liver and two gill samples were collected from each tank. The tissues were stored frozen before analysis. Samples were extracted as previously described (Nowak and Julli 1991).

The eluates were analysed using GLC (capillary column SE-30, carrier gas hydrogen, flow 2 ml min⁻¹, initial column temperature 60 °C, final column temperature 220 °C, column rate 50 °C min⁻¹, hold at final temperature 10 min, initial injector temperature 70 °C, final injector temperature 230 °C, injector rate 180 °C min⁻¹, hold at final temperature 2 min, detector temperature 350 °C). All residues were calculated based on tissue wet weights. The total endosulfan residue was calculated as the sum of alpha endosulfan, beta endosulfan and endosulfan sulphate.

Results of chemical analysis of endosulfan residues were obtained for fish kill cases held in the Land and Environment Court of New South Wales. The results from two fish kill cases were used: no. 50001 (EPA (Environment Protection Authority) versus Ron Harris 1992) and no. 50143 (SPCC (State Pollution Control Commission) versus