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SUSCEPTIBILITY OF FLUVALINATE AND ESFENVALERATE ON ADULT TAREK *Alburnus tarichi* (Güldenstädt 1814)

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ABSTRACT

Fluvalinate and esfenvalerate are pyrethroid insecticides and are used to control insect pests. These pesticides can enter the aquatic environment in different ways after use, causing toxicity. Tarek is a fish of the Cyprinidae family native to the Lake Van basin in Turkey. This study was conducted to determine the acute toxicity of fluvalinate and esfenvalerate to adult tarek. In the bioassays, tarek with an average length of 20.6±1.2 cm and an average weight of 93.9±14.0 g were used for fluvalinate, while an average length of 19.7±1.2 cm and an average weight of 85.5±16.6 g were used for esfenvalerate. Fish were exposed to fluvalinate using the semi-static test method, and esfenvalerate using the static test method. The toxicity tests were performed under the natural photoperiod. The concentrations used for fluvalinate were 0.15, 0.30, 0.45, 0.60 µg L^{-1}, while for esfenvalerate they were 0.33, 0.67, 1.00, 1.34 µg L^{-1}. The tests were performed at 13±1 °C for 96 hours with dechlorinated tap water. At the end of the toxicity tests, the mean lethal concentration (LC_{50}) of fluvalinate after 96 hours was determined to be 0.338 (0.230-0.477) µg L^{-1} and the above concentration of esfenvalerate was determined to be 0.475 (0.293-0.640) µg L^{-1} for adult tarek. Tarek exposed to fluvalinate and esfenvalerate showed toxic effects throughout the test, such as splashing, vigorous and then slower swimming, loss of balance, increased respiratory rate and contraction. Consequently, fluvalinate and esfenvalerate were found to be highly toxic to tarek and therefore the concentration of fluvalinate should not exceed 0.003 µg L^{-1}, and that of esfenvalerate should not exceed 0.005 µg L^{-1} in freshwater environments where tarek lives.

Keywords:
Cyprinidae
Pyrethroid insecticides
LC_{50}
Toxicity

How to Cite

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INTRODUCTION

Recently, the use of organophosphorus insecticides has shifted in favour of pyrethroid insecticides because of increasing reports of toxicity of organophosphorus insecticides in surface waters and lower toxicity of pyrethroids in mammals. Nowadays, pyrethroids are widely used in agriculture as well as in areas such as domestic use and veterinary medicine. Although these compounds are acutely less toxic to humans than organophosphorus insecticides, they are extremely toxic to fish and invertebrates in non-target waters (Brander et al., 2012). Pyrethroids are neurotoxic insecticides. They are among the most potent insecticides known. They are potentially toxic pollutants and cause significant direct risks in aquatic ecosystems (El-Sayed et al., 2007). Pyrethroids can enter the aquatic environment through spray drift or wash-off from land. Fluvalinate and esfenvalerate are among the pyrethroids (Medeiros et al., 2013). Fluvalinate is used to control insects and mites on potatoes and wheat, while esfenvalerate is used to control insect pests, especially on apples, peaches, cotton, and almonds. Fluvalinate and esfenvalerate are considered highly toxic to non-target organisms such as fish and aquatic invertebrates (EPA, 2005; EFSA, 2010; Rosa et al., 2016). The toxicity of fluvalinate and esfenvalerate to fish and aquatic invertebrates has been studied by many researchers (Kamalaveni et al., 2001; Werner et al., 2002; Denton et al., 2003; Wheelock et al., 2005; Forbes and Cold, 2005; Brander et al., 2012; Rosa et al., 2016).

Fish occupy an important place among aquatic organisms. In biological early warning systems, fish are often used as bioindicators of environmental stress. Therefore, fish bioassays are commonly used as experimental indicators to determine acute toxicity (Neelima et al., 2016; Kankaya and Kaptaner, 2017). Tarek is a cyprinid species endemic to the Lake Van watershed. It is consumed fresh and salted in settlements around Lake Van (Kankaya and Ünsal, 2018). The fish migrate to the rivers flowing into the lake in May-June to reproduce. The fish that have reproduced return to the lake. Hatched larvae also return to the lake after remaining in the river for some time (Kankaya and Kaptaner, 2014). During the reproductive migration of fish, it is inevitable that the fertilized eggs during incubation and the larvae when they return to the lake are exposed to possible toxic substances that mix with the river (Kankaya and Ünsal, 2018). This study was conducted to determine the acute toxicity of fluvalinate and esfenvalerate to sexually mature tarek *Alburnus tarichi* (Güeldenstäedt 1814), which may be mixed into streams by agricultural applications.

MATERIALS AND METHODS

Fish samples were collected from the natural environment in the Karasu River (38° 35' 22.17" N, 43° 13' 33.95" E), which flows into Lake Van, using a cast net. Fish were captured in May-June as they moved into the river for brood migration. Fish transported by oxygen-assisted thermos flasks were reared in fibreglass tanks (R90 h70) at appropriate densities. Dechlorinated, aerated tap water was used. A 7-day wait was made for the fish to acclimate to the new environment. During this time, the fish were maintained regularly. All procedures were performed with the approval of the Animal Experimentation Ethics Committee of Van Yuzuncu Yil University (Decision number: 2019/11).

The pesticides, Mavrik 2F 240 g L^{-1} Fluvalinate, Sungold 20 EC 200 g L^{-1} Esfenvalerate were purchased from a commercial company. Stock solutions of both chemicals were prepared. For toxicity tests, tarek with an average length of 20.6±1.2 cm and an average weight of 93.9±14.0 g were used for fluvalinate, while an average length of 19.7±1.2 cm and an average weight of 85.5±16.6 g were used for esfenvalerate. Fish were exposed to fluvalinate using the semi-static test method, and to esfenvalerate using the static test method. The fluvalinate test was continued by renewing the ambient water and chemicals at 24-hour intervals. Tests were conducted in fiberglass tanks (R90 h70) with 7 fish per application. Bioassays were performed under natural photoperiod. Tests were performed at 13±1 °C with dechlorinated tap water. The bioassays were performed for 96 h with two replicates (Ünsal, 1998; Çetinkaya, 2010). The concentrations of chemicals used in this study were determined considering previous toxicity tests (Bradbury and Coats, 1989; Lozano et al., 1992; Wheelock et al., 2005). The concentrations of fluvalinate and esfenvalerate tested were as follows: 0.00, 0.15, 0.30, 0.45, 0.60 µg L^{-1} and 0.00, 0.33, 0.67, 1.00, 1.34 µg L^{-1}, respectively. The chemicals were added to the water containing the fish by adding the stock solutions previously prepared. At the beginning of the bioassays, the values of temperature, dissolved oxygen, pH, electrical conductivity, total alkalinity and total hardness of the water were determined. During the test, temperature, dissolved oxygen, and pH values were recorded daily (Anonymous, 1995).

The LC_{50} value was calculated based on the number of dead and living fish determined over time during the bioassays (Ünsal, 1998; EPA, 2002; Çetinkaya, 2010). LC_{50} values and 95% confidence limits were calculated using a computer program with the probit analysis method.

RESULTS

The change of quality criteria of the water used in the bioassay is given in Table 1. The LC_{50} values calculated from the number of dead and living fish determined depending on time in the toxicity tests where tarek was exposed to fluvalinate and esfenvalerate are given in Table 2.

Table 1. The change of quality criteria in the water used in the toxicity test

Water quality criteria	Value
Temperature (°C)	13 ± 1
Salinity (‰)	0.4 ± 0.1
pH	8.57 ± 0.4
Dissolved oxygen (mg L^{-1})	6.41 ± 0.14
Dissolved oxygen saturation (%)	68 ± 5
Electrical conductivity (µS cm^{-1})	733
Total hardness, $CaCO_3$ (mg L^{-1})	354
Total alkalinity, $CaCO_3$ (mg L^{-1})	552

Table 2. The 96-hour LC_{50} values and their confidence limits determined for tarek in the fluvalinate and esfenvalerate toxicity test

Chemicals	Hours	LC_{50} (µg L^{-1})	LC_{50} (µg L^{-1}) 95% confidence limits
Fluvalinate	96	0.338	0.230 – 0.477
Esfenvalerate	96	0.475	0.293 – 0.640

Fluvalinate and esfenvalerate 96-hour LC_{50} values for mature tarek were determined as 0.338 (0.230–0.477), 0.475 (0.293–0.640) µg L^{-1}, respectively. 96-hour LC_{50} values and confidence limits determined after fluvalinate and esfenvalerate toxicity tests using different freshwater fish species and *Alburnus tarichi* are given in Tables 3 and 4.

Table 3. Fluvalinate 96-hour LC_{50} values at different purity levels and confidence limits for various other freshwater fish species (Anonymous, 2019a and present study)

Fish species tested	Fluvalinate formulation (%)	LC_{50} (µg L^{-1})	Confidence limits
Lepomis macrochirus	93.1	0.9	0.7 – 1.1
Lepomis macrochirus	24.9	2.1	1.4 – 3.1
Lepomis macrochirus	93.0	2.6	1.7 – 6.3
Lepomis macrochirus	93.2	2.7	–
Cyprinus carpio	93.1	4.8	3.4 – 6.6
Cyprinus carpio	–	13	–
Oncorhynchus mykiss	93.1	2.9	2.3 – 3.6
Oncorhynchus mykiss	24.9	8.8	–
Alburnus tarichi	Mavrik 2F 240 g L^{-1}	0.338	0.230 – 0.477

Table 4. Esfenvalerate 96-hour LC_{50} values at different purity levels and confidence limits for various other freshwater fish species (Anonymous, 2019b and present study)

Fish species tested	Esfenvalerate formulation (%)	LC_{50} (µg L^{-1})	Confidence limits
Lepomis macrochirus	–	0.44	0.41–0.47
Lepomis macrochirus	84	0.31	0.25–0.40
Oncorhynchus mykiss	44.4	0.07	0.057–0.087
Pimephales promelas	100	0.25	–
Pimephales promelas	–	0.22	0.18–0.27
Pimephales promelas	–	0.23	0.20–0.27
Pimephales promelas	–	0.32	0.26–0.40
Alburnus tarichi	Sungold 20 EC 200 g L^{-1}	0.475	0.293–0.640

Table 3 shows that the fluvalinate LC_{50} values given by other studies are higher than the values found for *Alburnus tarichi*. It can be said that *Alburnus tarichi* has a susceptibility to fluvalinate compared to the other fish given in the Table. Table 4 shows that the LC_{50} values of esfenvalerate determined in other fish are similar to those of *Alburnus tarichi*, therefore it can be said that the toxicity of esfenvalerate to *Alburnus tarichi* is quite high.

DISCUSSION

The 96-hour LC_{50} values determined in various fish species exposed to different pyrethroids have been reported in the results of many studies. Fish and chemicals used in the tests and obtained LC_{50} values have been reported as follows: 16.7 µg L^{-1} for tau-fluvalinate in *Danio rerio* (Jia et al., 2015), 5.99 µg L^{-1} for alpha-cypermethrine in *Oreochromis niloticus* (Sarıkaya, 2009), 7.2 nmol L^{-1} for fenvalerate in *Menidia menidia* and 803 nmol L^{-1} for permethrin in *Oncorhynchus mykiss* fish (Haya, 1989), 121.38 µg L^{-1} for fenpropathrin in *Alburnus mossulensis* (Banaee et al., 2014). In addition to these, 48-hour LC_{50} values of 1.17 and 1.70 µg L^{-1} have been reported for larvae and fry of *Oreochromis niloticus* fish exposed to deltamethrin (Karasu Benli et al., 2009). When the results obtained from studies on aquatic organisms with different pyrethroids were compared with the toxicity of fluvalinate and esfenvalerate to tarek, it was revealed that these two pesticides were highly toxic to tarek, although there were some variations.

Tarek, exposed to fluvalinate and esfenvalerate, showed toxic effects throughout the test, including splashing, vigorous and then slower swimming, loss of balance, increased respiratory rate, and contraction. Symptoms associated with the onset of death following acute exposure of fish to pyrethroids include hyperactivity, loss of balance, loss of herding behavior, increase in

respiratory rate, and convulsions, especially in the gill and jaw region (Haya, 1989). Loss of appetite, increased mucus secretion, increased abnormal behavior, surface swimming, and vertical swimming were found to be the main changes in *Alburnus mossulensis* fish exposed to sublethal high concentrations of fenpropathrin (Banaee et al., 2014). Fluvalinate and esfenvalerate used in this study were also pyrethroids and therefore showed similar results to the response table reported for pyrethroid insecticides in other studies.

CONCLUSION

Fluvalinate and esfenvalerate appear to be highly toxic to tarek. Fluvalinate should not exceed 0.003 µg L^{-1} and esfenvalerate should not exceed 0.005 µg L^{-1} in freshwater environments where tarek lives.

OSJETLJIVOST NA FLUVALINAT I ESFENVALERAT NA ADULTNOM BİSERNOM CİPLU, *Alburnus tarichi* (Güldenstädt, 1814)

SAŽETAK

Fluvalinat i esfenvalerat su piretroidni insekticidi koji se koriste za suzbijanje insekata. Ovi pesticidi nakon uporabe mogu ući u vodeni okoliš na različite načine, uzrokujući toksičnost. Biserni cipal je riba iz obitelji Cyprinidae porijeklom iz bazena jezera Van u Turskoj. Ovo je istraživanje provedeno kako bi se odredila akutna toksičnost fluvalinata i esfenvalerata na adultog bisernog cipla. U biološkim testovima za fluvalinat je korišten biserni cipal prosječne duljine 20,6±1,2 cm i prosječne težine 93,9±14,0 g, dok je za esfenvalerat korišten prosječne duljine 19,7±1,2 cm i prosječne težine 85,5±16,6 g. Ribe su bile izložene fluvalinatu semistatičkom test metodom i esfenvaleratu statičkom test metodom. Ispitivanja toksičnosti provedena su u prirodnom fotoperiodu. Koncentracije korištene za fluvalinat bile su 0,15, 0,30, 0,45, 0,60 µg L^{-1}, dok su za esfenvalerat korištene 0,33, 0,67, 1,00, 1,34 µg L^{-1}. Ispitivanja su provedena na 13±1 °C tijekom 96 sati s dekloriranom vodom iz slavine. Na kraju testova toksičnosti, srednja letalna koncentracija (LC_{50}) fluvalinata nakon 96 sati utvrđena je na 0,338 (0,230-0,477) µg L^{-1}, a gornja koncentracija esfenvalerata je određena na 0,475 (0,293-0,640) µg L^{-1} za odrasle biserne ciple. Biserni cipal izložen fluvalinatu i esfenvaleratu pokazao je toksične učinke tijekom cijelog testa, kao što su prskanje, žustro pa sporije plivanje, gubitak ravnoteže, povećana brzina disanja i kontrakcija. Stoga je utvrđeno da su fluvalinat i esfenvalerat vrlo toksični za ovu vrstu i stoga koncentracija fluvalinata ne bi smjela prijeći 0,003 µg L^{-1}, a koncentracija esfenvalerata ne bi smjela prijeći 0,005 µg L^{-1} u slatkovodnim sredinama u kojima biserni cipal živi.

Ključne riječi: Cyprinidae, piretroidni insekticidi, LC_{50}, toksičnost

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