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Biodegradability, toxicity, and photochemical treatment of a textile surfactant

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ABSTRACT

This study investigates the biodegradability, ecotoxicity, and photochemical treatment of a segregated textile bath discharge carrying a commonly applied nonionic surfactant. The wastewater having a total COD of 1,085 mg/L is completely soluble in nature. The COD distribution of this wastewater at different molecular size cut-off levels shows a fluctuating nature. Around 40% of the COD originates from a molecular weight cut-off range less than 30 kDa fraction. The wastewater is observed to contain an initially inert soluble COD of about 20 mg/L and the residual COD level achieved after passing it from aerobic biological treatment is only 40 mg/L indicating high biodegradability. After 120 min of UV-C application similar TOC removal efficiencies of 35 and 42% are obtained at pH 4 and pH 7, respectively. Photochemical treatment does not yield significant TOC removals at pH 11. The raw discharge is highly inhibitory toward *Vibrio fischeri*. 60 min of UV-C application lowered ecotoxicity toward *V. fischeri*.

Keywords: Inert COD; Photochemical treatment; Surfactant; Textile bath discharge; Ecotoxicity

1. Introduction

Today's understanding of industrial pollution control focuses mainly on waste minimization at source. As a result evaluating the auxiliary chemicals in terms of their biodegradability and ecotoxicity gains importance. Since a part of these auxiliaries are discharged into environment after being involved in the production, they are encountered with wastes. A large spectrum of auxiliary chemicals having variable biodegradability and toxicities sometimes with carcinogenic, mutagenic, or teratogenic properties [1] are introduced during the production of textile goods. The intensive auxiliary inputs results in the generation of chemical laden textile effluents. Application of green chemistry to textile wet processing dictates the fact that reactants, end products, and wastes generated should have low toxicity to humans/environment and preferably be biodegradable [2]. In this respect, research activities on textile auxiliaries are intensified recently. Ecotoxicity and biodegradability of textile auxiliaries [3–13], substitution of textile chemicals [14–16], development of new production processes [17], the effect of textile auxiliaries on treatability [18],

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and investigation of certain treatment methods for the removal of various auxiliaries [19,20] are among them.

Different classes of nonionic and mixed nonionic/anionic surfactants (wetting agents) are applied during the preparation and finishing stages of textile processing. Surfactants can cause foam formation in environment and anomalies in the growth of algae [21]. In some cases, the biodegradation of the some nonionic surfactants is observed to generate more hydrophobic metabolites yielding a more toxic outcome than the parent product [1]. Since more than 500 unique nonionic surfactants are applied in textile wet mills [22], particular attention must be given to them.

Biological treatment is the most commonly applied treatment method for textile wastewaters. The biodegradability of auxiliaries dictates the level of organics (i.e. chemical oxygen demand; COD), that can be reached after passing the textile wastewater from biological treatment. The total COD, C_T, of raw wastewaters can be categorized into two main fractions: The inert COD and the biodegradable COD. As all the biodegradable organics are expected to be removed in a properly designed and well operated biological treatment plant, the outlet of such biological treatment systems does not contain any biodegradable COD. Particulate inert COD fractions leave the treatment facility by sludge wastage. On the other hand, the soluble inert COD of the raw wastewaters, S_I, bypasses the biological treatment system without being removed. Furthermore, residual microbial products, both in particulate and in soluble nature, are generated through the course of biochemical reactions. Similar to that of particulate inert COD fractions, particulate residual microbial products will be removed via sludge wastage, whereas soluble residual microbial products and the soluble inert COD of the raw wastewaters will present in the treated effluents. The soluble residual (inert) microbial products, S_P, and the inert COD of influent origin, S_I, all together dictates the level of achievable COD at the outlet of a biological treatment plant [23]. The sum of these two fractions gains importance in meeting the standards. While evaluating different auxiliary chemicals their ecotoxicity levels must be considered alongside their biodegradability. Ecotoxicity and biodegradability of auxiliaries can be used as a guide to select the proper chemical if there exists alternative additives performing the same function during the production.

Biodegradability (or inert COD) and ecotoxicity of segregated industrial effluents can be adapted to industrial wastewater management in a way to ease the evaluation based on technical and economical feasibility of pollution control alternatives. If a segregated industrial effluent contains elevated levels of soluble inert COD and/or high ecotoxicity, a specific treatment alternative such as AOP's [24–26], directly targeting the reduction of refractory organics and/or ecotoxicity, can be applied to this segregated effluent stream. By doing so, the problem related to the biodegradability and/or ecotoxicity of a relatively low volume of segregated effluent can be solved on rather economical grounds. Within the last five years substantial increase is observed in research activities dealing with treatment of industrial effluents by AOP's and their effects on ecotoxicity [4]. The application of partial specific treatment such as AOP's must be handled carefully as a part of the target contaminants might be transformed to unknown oxidation products with unknown ecotoxicities. Therefore, such partial treatability assessments must be supported with ecotoxicity tests. AOP processes are widely applied to deal with problematic textile effluents containing textile auxiliaries such as surfactants [27–42]. The advantage of treatment with UV can be enlisted as no sludge formation, simple operation, and no phase transfer problems [18]. Some segregated textile effluents contains H_2O_2. Due to this fact, it is beneficial to apply UV to get H_2O_2 enhanced treatment.

In this context, the aim of this manuscript is to investigate the biodegradability and ecotoxicity of a commonly applied nonionic textile surfactant. For this purpose, the segregated textile bath discharge carrying the surfactant is subjected to biodegradability experiments and ecotoxicity tests. The COD distribution of the segregated bath discharge at different molecular size cut-off levels is examined. Photochemical treatment of segregated wastewater by UV-C and the effect of treatment on ecotoxicity are also inquired.

2. Materials and methods

2.1. Segregated textile bath discharge bearing surfactant under investigation

The commercially applied surfactant was obtained from a local textile dyeing and finishing mill in Istanbul, Turkey and used as received. During production of viscose fabric, this surfactant was added in batchwise preparation operations prior to optical bleaching step and a segregated wastewater discharge that contains the surfactant was generated as given in Fig. 1. In the preparation stage where the surfactant was added, viscose fabric was processed at 95˚C at a pH of 10 for about 60–75 min. The surfactant was applied at a concentration of 8 g/L to the textile bath (i.e. 4% of fabric weight) and approximately 100% of the surfactant remained unfixed onto the textile material yielding a segregated wastewater containing all of the

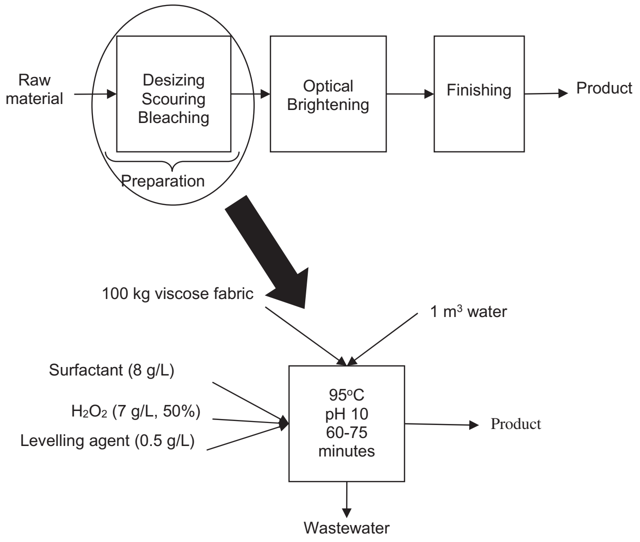

Fig. 1. Process flowchart.

added surfactant. Similarly, the other bath inputs, i.e. H_2O_2 and leveling agent were not attached onto the textile fiber. Therefore, to mimic the actual textile bath discharge where this textile auxiliary was added, a sample was prepared by dissolving 8 grams of surfactant, 7 grams of H_2O_2 (50%), and 0.5 grams of leveling agent in 1 L water. All the experiments were performed by using this sample.

Physicochemical and ecotoxicological characteristics of the surfactant are tabulated in Table 1. The surfactant has low acute oral toxicity toward rats. However, an assessment based on aquatic ecotoxicity must be conducted as the wastewater generated from this industry will be discharged to a nearby marine environment.

2.2. Molecular size distribution experiments

To support the conventional wastewater characterization, the COD distribution at different molecular size cut-off levels was examined by employing a series of filtration/ultrafiltration experiments on textile bath discharge. These experiments were performed in 400 mL-capacity cells (Amicon, Model 8400) under positive nitrogen gas pressure (0.4–2.5 bar) and continuous mixing. For that purpose, the synthetically prepared textile bath discharge was filtered through 1,200–1,600 nm (Millipore AP40), 450 nm (Durapore® HV, PVDF), and 220 nm (Durapore® GV, PVDF) cut-off filters under 0.7 atm pressure. During the ultrafiltration experiments, samples previously filtered through 220 nm membrane filters were subjected to ultrafiltration using 100, 30, and 10 kDa molecular size cut-off membranes under 3.7 atm pressure. Obtained permeates and retentates were analyzed and fractionated for COD.

2.3. Inert COD Experiments

The inert COD content of the textile bath discharge containing the surfactant under investigation was assessed by following an experimental procedure described in the literature [43]. Two aerated cylindrical batch reactors, one fed with the textile bath discharge itself and the other with glucose solution having the same dilution, were run. The volumetric capacity of the reactors was 3 L. The seed was obtained from a lab-scale fill and draw aerobic reactor operated under steady state conditions for about 40 d. This reactor was run with a food to micro-organism ratio of 0.6 mg COD (mg VSS d)$^{-1}$. A mixture of 50% glucose and 50% textile bath discharge sample was applied as the feed for this fill and draw aerobic reactor that was used to generate the seed. An initial biomass concentration of 50 mg VSS L^{-1} was sustained in both of the batch reactors (run with textile bath discharge and with glucose), where inert COD tests were performed. Such a low initial biomass is adopted to hinder the interference of the residual COD that can form from the endogenous respiration of initial inoculums. Aliquots removed periodically from the mixed liquor of each reactor (run with textile bath discharge and with glucose) were analyzed for soluble COD. To avoid erroneous results, a strict accounting was kept

Table 1
Characteristics of the surfactant as obtained from the product safety data sheet

Appearance	Cream colored granules with a characteristic odor
Ingredients	Ethoxylated alcohol mixture (non-ionic surfactant)
Density (g/cm^3)	0.93
pH	10–11 (1 g/L)
Solubility	Highly water soluble
Acute oral toxicity (LD$_{50}$^{a}, mg/kg)	>1,200

[a] Lethal dose causing 50% death in rats.

for all samples removed from the reactors, so that any water loss by evaporation was replaced with water prior to sampling. When a stable soluble COD plateau (where the threshold level was established as less than 3–5% variation between consecutive samples) was observed, the experiments were ceased. During the operation of batch aerobic reactors, the maintenance of the pH in the range of 7–8 (suitable for biological activity) was strictly followed. Nitrification inhibitor (Formula 2533TM, Hach Company) was added to all bioreactors in order to prevent any possible interference induced.

2.4. Photochemical treatment experiments

Photochemical treatment experiments were conducted using a 3,250 mL capacity batch stainless steel photoreactor (length 95 cm, width 6 cm) equipped with a 40 W low-pressure, mercury vapor sterilization lamp located at the center of the reactor in a quartz glass envelope. The incident light flux of the UV-C lamp at 253.7 nm and effective UV-C light path length were determined via H_2O_2 actinometry [44] as 1.44×10^{-5} einstein L^{-1} s^{-1} and 5.67 cm, respectively. During the experiments, segregated textile bath discharge was continuously circulated through the UV-C photoreactor using a peristaltic pump at a rate of 400 mL/min, corresponding to a hydraulic retention time of 8 min in the photoreactor. During a typical experimental run, the surfactant wastewater was fed to the photoreactor and a sample (at time $t = 0$) was taken. At this point, the reaction was initiated by turning on the UV-C lamp. About 30 mL sample aliquots were taken at regular time intervals for up to 120 min and analyzed for total organic carbon (TOC) and H_2O_2.

2.5. Acute ecotoxicity measurements

As the final receiving body of the treated effluent is sea, acute ecotoxicity of raw (untreated) and treated surfactant wastewaters were measured with a BioTox™ test kit (AboatoxOy, Finland), a commercial bioassay based on the inhibition of bioluminescence emitted by the *Vibrio fischeri* marine photobacteria in accordance with the ISO 11348-3 protocol [45]. The lyophilized bacteria were rehydrated according to the test protocol; then, 500 μL aliquots of the bacteria solution were pre-incubated for 15 min at 15˚C. After the initial bacteria luminescence had been measured, 500 μL of the diluted samples were added to the bacteria. The luminescence was again measured after the incubation time of 15 min at 15˚C on an Aboatox C110 (AboatoxOy, Finland) BioTox apparatus. The IC_{20}, $IC_{50,}$ and IC_{80} (the dilution ratio of samples that produces 20, 50, and 80% effect on *V. fischeri*, respectively) of raw and treated surfactant wastewaters were determined from different dilution ranges. Prior to the assay the pH of all samples was adjusted to 7.0 ± 0.2 with NaOH or H_2SO_4 solutions.

2.6. Analytical procedure

COD was determined by the closed reflux titrimetric method according to ISO 6060 [46]. Prior to COD analysis, H_2O_2 present in the samples was destroyed with enzyme catalase derived from *Micrococcus lysodeikticus* (Fluka) to prevent interference of H_2O_2 with the COD measurements. TOC was monitored on a Shimadzu V_{CPN} model carbon analyzer (combustion method) equipped with an autosampler. H_2O_2 in the samples was determined titrimetrically by employing the molybdate-catalyzed iodometric method [47]. All other analyses for conventional characterization were performed as defined in Standard Methods [48]. All experiments were conducted at room temperature. pH adjustments were made by NaOH or H_2SO_4 solutions and samples pH values were determined with an Orion 720+ model pH-meter. Filtrates of samples subjected to vacuum filtration by means of Millipore membrane filters with a pore size of 0.45 μm are defined as soluble fractions. All experiments were conducted at least in duplicate and repeated, until statistically indifferent results were obtained. Therefore, all experimental data/results reported herein are within the standard error range of all instrumental and wet analysis procedures used in this study.

3. Results and discussion

3.1. Conventional wastewater characterization and molecular size distribution

The textile bath discharge where the surfactant under investigation is added has a total COD of 1,085 mg/L (which is completely soluble in nature), TKN of 56 mg/L, TP of 1 mg/L, and a pH range of 10–11. The distribution of COD in terms of different molecular weight cut-offs is presented in Fig. 2. The results indicate that approximately 41% of the COD originates from a molecular weight cut-off range less than 30 kDa fraction. As can be seen from the figure a fluctuating profile of COD is obtained.

3.2. Inert COD

Table 2 tabulates the results of inert COD experiments obtained on the segregated textile bath, where

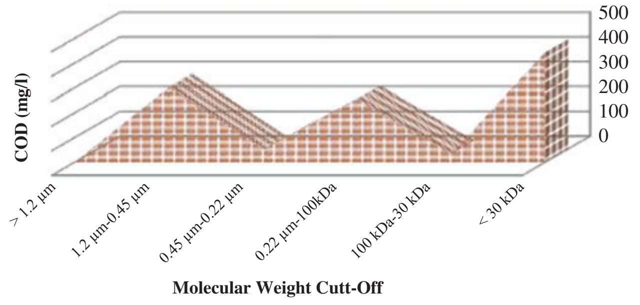

Fig. 2. COD based molecular size distribution of textile bath discharge.

the surfactant under investigation is introduced. The segregated bath discharge, completely soluble in nature, was assessed to contain an initially inert soluble COD of 22 mg/L. The residual COD level achieved after passing this bath discharge from aerobic biological treatment is only 40 mg/l indicating the highly biodegradable characteristic of the segregated textile effluent.

3.3. Photochemical treatability and its effect on ecotoxicity

TOC and H_2O_2 abatements observed during the photochemical treatment of surfactant wastewater at different pH values; pH 4 (acidic), pH 7 (neutral), and pH 11 (alkaline) are shown in Fig. 3(a) and (b), respectively.

As seen from Fig. 3(a), similar TOC removal efficiencies were obtained at pH 4 (35%) and pH 7 (42%) after the application of 120 min treatment. Such duration corresponds to the exhaustion of about 40% of the initially present H_2O_2 for both pHs (Fig. 3(b)). In case of pH 11, H_2O_2 consumption was rapid and high (% 71) for which TOC removal was limited to only 12%. Upon closer inspection of the TOC and H_2O_2 abatement profiles obtained at pH 11, it could be observed that practically no TOC removal (≈4%) and H_2O_2 consumption (≈1%) appeared during the first 25 min of photochemical treatment and both TOC and H_2O_2 removals increased thereafter. Under alkaline conditions, the amount of hydroperoxide ion (HO_2^-) increases due to H_2O_2 dissociation (pK_a of $H_2O_2 = 11.6$) [49]. HO_2^- ion on the one hand, reacts with non-dissociated molecules of H_2O_2, which leads to dioxygen and water (Eq. (1)), and on the other one absorbs incident radiation more effectively ($\varepsilon_{254} = 240\ M^{-1}\ cm^{-1}$) [50] than H_2O_2 (with the same quantum yield as H_2O_2). HO_2^- ion also undergoes photolysis giving $HO^{\bullet}$ and oxygen radical anion ($O^{\bullet -}$), but they react very quickly ($2 \times 10^{10}\ M^{-1}\ s^{-1}$) [51] giving back HO_2^-. Therefore, the instantaneous concentration of $HO^{\bullet}$ is lower than expected at alkaline reaction conditions.

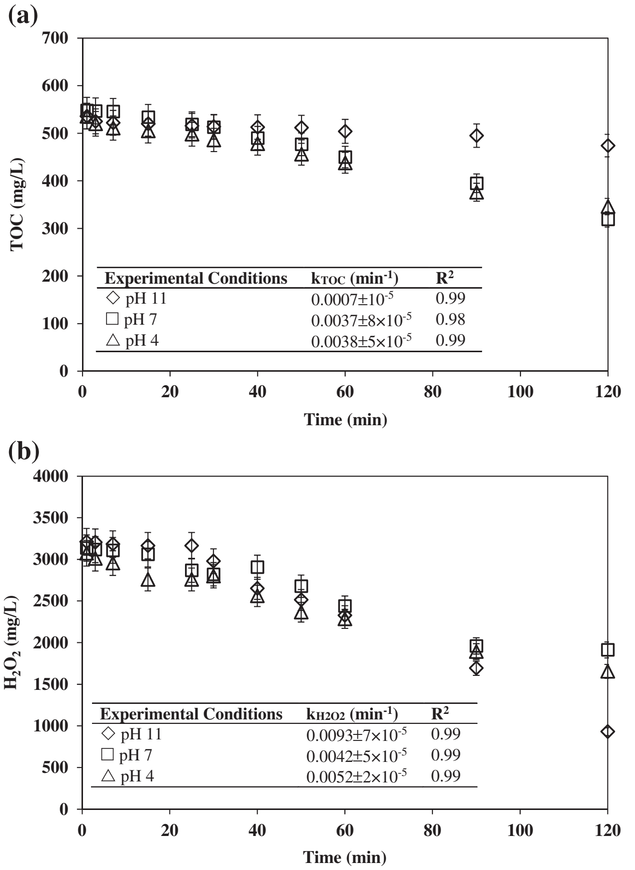

Fig. 3. TOC (a) and H_2O_2 (b) abatements obtained during the photochemical treatment of textile bath discharge at pH 11, pH 7, and pH 4.

$$H_2O_2 + HO_2^- \longrightarrow H_2O + O_2 + OH^- \tag{1}$$

Table 2
Inert COD fractions of textile bath discharge carrying the surfactant

COD S_T (mg/L)	Initially inert soluble COD S_I (mg/L)	Residual COD $S_R = S_I + S_P$ (mg/L)	S_R/S_T (%)
1,085	22	40	4

Under the studied treatment conditions, TOC removal and H_2O_2 consumption rates followed pseudo-first-order kinetics (see legend of Fig. 3 for calculated apparent first-order rate constants) with high determination coefficients ($R^2 \geq 0.98$). The calculated apparent first-order TOC removal rate constants (k_{TOC}, min^{-1}) followed the decreasing order; pH 4 = pH 7 (0.0037 min^{-1}) > pH 11 (0.0007 min^{-1}), indicating that the acidic or neutral pH values were required for removal of organics from the surfactant wastewater. On the contrary, H_2O_2 consumption rate constants were 1.7–2.2 times faster at basic pH ($k_{H_2O_2}$ = 0.0093 min^{-1} at pH 11) than at neural and acidic pHs ($k_{H_2O_2}$ = 0.0042 min^{-1} at pH 7 and $k_{H_2O_2}$ = 0.0052 min^{-1} at pH 4). Upon closer inspection of the H_2O_2 consumption profile obtained at pH 11, it could be observed that during the first 25 min of the reaction, practically no H_2O_2 utilization (only 47 mg/L) was recorded and a significant reduction in H_2O_2 values was obtained after a reaction period of 25 min. Hence, kinetics could only be established after this irradiation time of 25 min.

In order to determine how the initial pH influences ecotoxicity of raw surfactant wastewater, the pH of the samples were adjusted to acidic, neutral, and basic values (pH 4, 7, and 11) and the samples were allowed to stay at room temperature overnight. The *V. fischeri* toxicities (IC values) of untreated and photochemically treated surfactant wastewaters at different initial pH and treatment durations are tabulated in Table 3. It should be emphasized here that any H_2O_2 present in the samples was destroyed with enzyme catalase prior to ecotoxicity analysis in order to eliminate its false positive effect on the test results.

The original surfactant wastewater was highly inhibitory towards *V. fischeri*. The decreasing order of *V. fischeri* ecotoxicity of surfactant wastewater at the investigated initial pH values were found as follows; acidic pH (= 4) > neutral pH (= 7) ≈ basic pH (= 11). As can be followed from Table 3, a general increase in IC values was observed for 60-min treated samples indicating the photochemical oxidation products formed at this particular treatment time were less toxic than the original surfactant for all the initial pH settings. However, as the oxidation progressed a decrease in IC values was observed for all experiments conducted at the investigated pH values. The ecotoxicity profiles obtained during photochemical treatment, demonstrated the formation and a subsequent degradation of toxic oxidation products towards *V. fischeri*. The same toxicity profile (an increasing trend at the initial stages and then a drop) during the $HO^{\bullet}$-based treatment of alkylphenol ethoxlate type nonionic surfactant has also been reported in other relevant studies [52,53]. For example, Karci et al. [52] comparatively studied H_2O_2/UV-C oxidation and UV-C photolysis of nonylphenol decaethoxylate (NP-10) with respect to *V. fischeri toxicity*. It was concluded that H_2O_2/UV-C process can be more safely applied for the removal of aqueous NP-10 than UV-C photolysis when toxicity is concerned. In another study [53], where peroxymonosulfate (PMS)/UV-C oxidation of octylphenol ethoxylate was investigated, a prompt increase in relative inhibition followed by a sharp decrease after 10 min PMS/ UV-C treatment was observed.

Table 3
Experimentally obtained IC values (%, v/v) for the raw (untreated) and photochemically treated textile bath discharge at different initial pHs

	Raw	60 min treated	90 min treated
Basic pH (=11)			
IC_{20}	0.6	0.9	0.8
IC_{50}	0.9	1.5	1.3
IC_{80}	2.2	4.0	3.3
Neutral pH (=7)			
IC_{20}	0.5	0.8	0.3
IC_{50}	0.8	1.3	0.5
IC_{80}	2.0	3.5	1.3
Acidic pH (=4)			
IC_{20}	0.1>	1.3	0.7
IC_{50}	0.1>	2.0	1.1
IC_{80}	0.1>	5.3	2.9

4. Conclusions

The concluding remarks obtained from this study that investigates the biodegradability, ecotoxicity, and photochemical treatment of segregated textile bath discharge carrying a commonly applied nonionic surfactant are summarized below.

Completely soluble wastewater has a total COD of 1,085 mg/L. The COD distribution at different molecular size cut-off levels indicates that approximately 40% of the COD originates from a molecular weight cut-off range less than 30 kDa fraction.

Inert COD experiments show almost completely biodegradable nature of the effluent. On the other hand, the discharge is found to be highly inhibitory towards *V. fischeri*.

UV-C application does not yield significant TOC removals at pH 11. Acidic or neutral pH ranges are required for the removal of organics from the wastewater via UV-C.

An improvement in ecotoxicity towards *V. fischeri* is observed after 60 min of UV-C application.

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