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Project Summary

An Investigation of Selected Dye Carriers Used in Commercial Dyeing of Hydrophobic Fibers

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This project was initiated to investigate potential toxicities of dyebath discharges from atmospheric dyeing of 100% polyester fabric with three commercial dye carriers: trichlorobenzene (TCB), biphenyl (BP), and o-phenyl phenol (OPP). First, criteria were established to rank the priority of all commercial dye carriers, based on known toxicities, volumes of consumption, etc. Commercial dyeing of polyester fiber/fabric was simulated in the laboratory, using a commercial disperse dye, other dyebath additives, and the selected dye carriers.

Biological testing and chemical analyses were carried out on the discharges of these dyebath systems. Bioassays included 1) Ames Salmonella/microsome mutagenesis assay, 2) acute *in-vivo* mouse quantal assay and, 3) aquatic ecological assay with *Daphnia magna.* Analytical procedures were established for both qualitative identification and quantitative determination of key dyebath components.

In-depth studies were also carried out on two dyebath systems (TCB and BP) to identify species responsible for the observed toxicity and to delineate possible chemical and/or biological interactions between the dyebath components.

The TCB system was also investigated to demonstrate that the toxicity of these dyebath discharges can be reduced significantly by increasing dyeing efficiency. The dyeing process was carefully evaluated to determine the dye and carrier contents necessary to achieve maximum dyebath efficiency.

This Project Summary was developed by EPA's Industrial Environmental Research Laboratory, Research Traingle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

The Clean Water Act of 1977 identified 65 toxic compounds and classes of toxic compounds which are subject to effluent limitations and expanded the spectrum of pollutant parameters to receive attention in point source discharges to include potentially toxic pollutants. Within these 65 compounds and classes, specific elements or compounds have been identified as priority pollutants. Included in this priority list of 129 compounds are 13 metals, 114 organic compounds, cyanide, and asbestos.

Wastewater guidelines for the textile industry were developed in 1977, based on studies sponsored jointly by EPA and the industry. The water use and wastewater discharge statistics for the textile industry were reported in detail by Sverdrup and Parcel and Associates, Inc. in a study conducted for EPA. This study indicates that the dyeing step in the finishing of woven and knitted fabrics and in carpet finishing is responsible for most of the waste volume and contributes substantially to the waste load in the finishing process. Color is an obvious adverse characteristic of the wastewater, along with high levels of dissolved solids. Carriers used in dyeing of polyester (and other hydrophobic fibers) contribute significantly to the biochemical oxygen demand (BOD). Carriers which are used to accelerate the dyeing of hydrophobic fibers with disperse dyes (and sometimes

other classes of dyes) are of particular concern in the dyeing waste stream since some of them appear as specific chemicals on EPA's Priority Pollutant List.

The present study has focused on the laboratory investigation of the toxicity of major dye carriers currently used in the commercial dyeing of polyester fiber/fabric. The study includes development of analytical techniques and systematic biological evaluation, necessary to identify and quantify toxic species in these systems. The study was also intended to provide information which can be used to eliminate or to reduce toxic substances in textile industry waste discharges.

The systematic approach taken to carry out the above objectives included:

1. Establishing priority ranking of current commercial dye carriers for polyester fiber/fabric, based on available data on current consumption and toxicity of these chemicals.
2. Biological and chemical screening of the three top-ranked carrier systems, according to the order of the priority established.
3. In-depth biological and chemical studies of the first two toxic systems identified in the above screening studies, with the objectives of identifying the species responsible for the observed toxicity and delineating possible interaction (synergistic or antagonistic) between species.
4. Investigating means of eliminating or reducing toxicity of these systems, by varying dyebath contents, dyebath conditions, and/or dyeing procedures.

Three commercial dye carriers, trichlorobenzene (TCB), biphenyl (BP), and o-phenyl phenol (OPP), were selected based on the priority ranking established. A commercial dye, disperse Yellow-23 (Y-23), and other dyebath additives were used. A typical commercial atmospheric dyeing process was simulated in the laboratory to carry out all dyeing operations. Biological evaluations and chemical analyses were carried out on the spentbath liquor from each screening dyebath. Bioassays included 1) Ames Salmonella/microsome mutagenesis assay, 2) acute *in-vivo* mouse quantal assay and, 3) aquatic ecological assay with *Daphnia magna.*

Various analytical techniques were used for qualitative and quantitative analyses of key dyebath components in the spentbaths and on dyed fabrics. An analytical procedure using a computer-assisted infrared (IR) spectrophotometer was established for qualitative identification of key dyebath components. Analytical procedures employing a high-performance liquid chromatography (HPLC) and an ultraviolet/visible spectrophotometer (UV/VIS) were also established for quantitative analyses of carriers and dye.

In-depth studies of two carrier systems (TCB and BP) were carried out to identify the dyebath component(s) responsible for the observed toxicities. The potential of reducing the toxicity of these dyebath systems by varying dyeing conditions and initial dyebath compositions was also investigated.

Conclusions

Toxicity screening of spentbath samples from laboratory-simulated atmospheric dyeing of 100% polyester fabric with a commercial disperse dye (Yellow-23) and commercial dye carriers (TCB, BP, and OPP) indicated that all three carrier systems are extremely toxic to *Daphnia magna* assay. Median effective concentrations, EC_{50}, for the 48-hr test were 25.5, 9.28, and 0.91%, respectively, for the TCB, BP and OPP carrier systems. However, results of Ames Salmonella/microsome mutagenesis assay and acute *in-vivo* mouse quantal assay were negative for all three carrier systems. Results of both bioassays and analyzed concentrations of dye and carriers for all three systems screened are shown in Table 1.

In-depth studies, carried out on the TCB and BP carrier systems, showed that toxicity in these two systems was due mainly to the dye (Yellow 23) and the carrier (TCB or BP); no other dyebath additives were found to have any significant toxicity. Furthermore, the toxicity observed in these systems was found to be the additive effect of the dye and the carrier. The OPP system (the most toxic to *Daphnia magna*) was not studied in depth because of project budgetary constraints.

A material balance made around the dyeing process showed that, in all three systems (TCB, BP, and OPP), a significant amount (more than 50%) of the original carrier content in the dyebath was lost, through volatilization, during dyeing. This may have an adverse impact on the air environment.

Studies varying dyeing time and original carrier content indicate that dyeing conditions can be optimized to minimize the amount of residual dye and carrier in the wastewater. This is one potential technique for reducing the toxicity of these wastewaters.

The present study has developed and established a basis for analytical techniques which can be applied to relatively simple textile waste systems, such as dyebath discharges. It has also demonstrated that the systematic approach of biological and chemical screening and in-depth investigation effectively identifies toxic species and evaluates overall toxicity of these systems. However, successful applications of the techniques and the approach to a more complex textile waste discharge remains to be demonstrated.

Table 1. *Summary of Screening Studies*

	TCB System	*BP System*	*OPP System*
Spentbath Contents:			
Dye (Yellow-23), mg/l	*5.32*	*5.34*	*2.98*
Carrier, mg/l	*63.0(1,2,4, TCB)*	*41.9(BP)*	—[a]
Bioassay Results:			
Ames (w/ and w/o liver microsomal enzyme)	*Negative*	*Negative*	*Negative*
Mouse Quantal Assay	*Nontoxic*	*Nontoxic*	*Nontoxic*
Daphnia magna Assay			
EC_{50}, %			
24 hr [b]	*31.2(24.3-38.1)*	*20.45(8.16-51.25)*	*0.91(0.70-1.12)*
48 hr [b]	*25.5(19.2-32.7)*	*9.28(7.74-10.83)*	*0.91(0.65-1.18)*

[a] *Not analyzed.*
[b] *Values in parentheses are 95% confidence intervals.*

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The complete report, entitled "An Investigation of Selected Dye Carriers Used in Commercial Dyeing of Hydrophobic Fibers," (Order No. PB 83-247 569; Cost: $13.00, subject to change) will be available only from:
National Technical Information Service
5285 Port Royal Road
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The EPA Project Officer can be contacted at:
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