

## ABSTRACT

E. ANDY KUBIAK. The Feasibility of Wastewater Reuse at the National Spinning Company, Inc., Washington, North Carolina. (Under the direction of Dr. FRANCIS A. DIGIANO)

The reuse of municipal wastewater is becoming increasingly popular in many areas of the United States, especially those areas where clean, fresh water is in high demand. Reclaimed wastewater can be a valuable resource in applications where less than potable water quality is sufficient. The City of Washington, North Carolina and the National Spinning Company, Inc. are considering a unique wastewater reuse scheme. Upon upgrading of the wastewater facility at the City of Washington with a denitrification filter, it is proposed that 1.3 million gallons per day (mgd) of tertiary treated wastewater be routed for use as process water in textile dyeing operations. The reclaimed wastewater would be stored at two abandoned City of Washington clearwells prior to use by National Spinning Company.

A pilot-plant was constructed at the University of North Carolina at Chapel Hill to simulate the City of Washington treatment plant upgrade and the present process water treatment scheme at the National Spinning Company (a groundwater source). Several large grab samples of City wastewater were collected and processed through the pilot-plant. The reclaimed water was analyzed to determine the feasibility for use as process water.

The results indicated that the reclaimed wastewater is of very high quality based on conventional measures (COD, BOD, and TOC); however, regrowth of microorganisms during storage of the reclaimed water is expected. The addition of a strong oxidant would likely reduce this regrowth. In addition, the reclaimed wastewater has the potential to stain the National Spinning Company fabrics. Applying a standard treatment scheme of coagulation-flocculation-sedimentation to the stored water appears to significantly reduce the staining; however, some staining potential is still evident.

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## CHAPTER ONE

### INTRODUCTION

#### Background

The City of Washington, North Carolina and National Spinning Company, Inc. are considering a unique wastewater reuse scheme. Upon upgrading of the wastewater treatment facility at the City of Washington with a denitrification filter (Tetra Engineered Systems, Pittsburgh, PA), it is proposed that 1.3 mgd of the 2.5 mgd tertiary treated wastewater be routed for use as process water in textile dyeing operations. Figure 1-1 displays a schematic of the proposed reuse plan. The treated wastewater would be pumped 1,900 feet to existing effluent storage reservoirs (the two clear wells of the abandoned Jordan water treatment facility for the City of Washington) and then pumped a total distance of about 3,400 feet to the existing water treatment facility at the National Spinning Company, Inc.

Clearly, the proximity of the City's wastewater treatment facility and the industry and existing storage reservoirs make this proposal attractive. Moreover, the final point of wastewater discharge for a large percentage of the City's flow will be shifted from Kennedy Creek to the Tar River. This shift will provide a larger dilution factor and possibly relieve the City from having to construct a new outfall of its own to the Tar River.



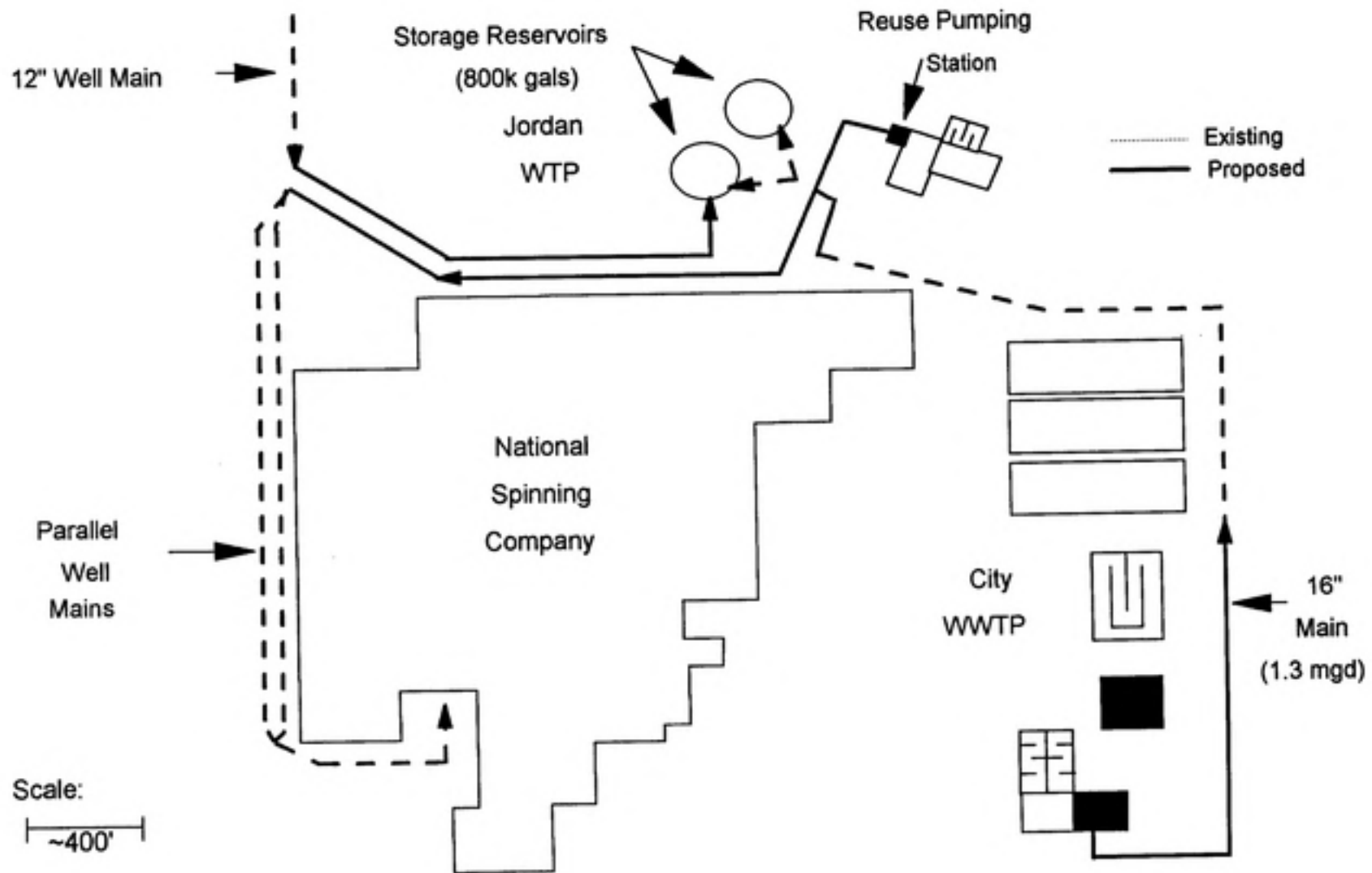


Figure 1-1. Schematic of Proposed Reuse Plan

The reclaimed water would be used in yarn dyeing operations. At present the National Spinning Company uses a groundwater supply for process water. This water is not used as a source of potable water at the facility and is, therefore, not regulated. Consequently, there is no concern at the facility about employee exposure or contact with the present process water. The conversion of this water to supply reclaimed wastewater will likely heighten employee awareness at the facility as well as require the National Spinning Company to regulate this water source.

The proposed wastewater reuse scheme would be a unique situation in North Carolina and would require close cooperation with State regulating departments. Presently, the regulation of potable water sources in North Carolina is governed by the Division of Health Services while the regulation of wastewater discharge is governed by the Division of Environmental Management. This situation would, therefore, be a special case in the State of North Carolina and would need to be handled as such.

#### Research Objectives

The research objectives of this project were:

- to determine if the reclaimed wastewater quality meets process water requirements at the National Spinning Company, Inc.

- to compare the quality of the reclaimed water to the current groundwater supply used by National Spinning Company, Inc.

## CHAPTER TWO

## REVIEW OF LITERATURE

Wastewater Reuse

**Background.** The reuse of treated wastewater as a water resource has been practiced throughout the world for centuries. In the United States, reclaimed wastewater has been used to supplement shortages in available water resources and as a means of pollution abatement. Reclaimed water can replace potable water for many uses that do not require water of such high quality, including: crop and landscape irrigation; non-potable urban uses; such as residential lawn irrigation and toilet flushing; industrial cooling and process water; and groundwater recharge.

Several states have been at the forefront of wastewater reuse technology. In California, water shortages have dictated the need for water conservation and reuse. As early as 1932, the Golden Gate Park in San Francisco began using reclaimed water for large-scale, landscape irrigation. Since 1976, reclaimed water has been injected directly into groundwater aquifers to prevent salt water intrusion in the coastal areas and as a means of supplementing potable water supply sources ( Crook, et. al., 1993).

Over the past twenty years, the State of Florida has implemented a number of reuse projects. The driving force in Florida has been to limit effluent discharges into low flowing,

shallow surface water sources (York, et. al., 1991). Also, population surges in coastal areas have resulted in some salt water intrusion of the local drinking water aquifers. Florida has developed programs that encourage the reuse of reclaimed wastewater throughout the state. In 1977, St. Petersburg began using a dual water system in which reclaimed water was supplied for industrial use and irrigation of golf courses, parks, and individual home sites (Crook, et. al., 1993). Reclaimed water has also been used extensively in Florida for crop irrigation as well as other non-potable residential uses (York, et. al., 1991).

In North Carolina, wastewater reuse has not been as common due to the abundance of water supplies. However, tighter regulations on wastewater discharges may lead communities to consider the reuse of wastewater effluent to offset demands for non-potable water. This is particularly true for cases where the existing water supply is limited, is of poor quality, or where the reduction of treated effluent discharge may result in reduced pollutant loadings on receiving streams (Miles, et. al., 1993).

**Industrial Applications.** The primary concern of this study is in the use of reclaimed wastewater for industrial applications. The most common industrial use of reclaimed water has been as a source of cooling water. The use of reclaimed water has been used as cooling tower make-up and as a primary source of once-through cooling water (EPA, 1992). The use of municipal wastewater for industrial cooling systems has been practiced by a variety of

industries. The Bethlehem Steel plant in Sparrows Point, Maryland, has used secondary effluent for once-through and direct metal cooling since 1942. In Oklahoma, Farmland Industries has been using the City of Enid's municipal wastewater effluent since 1974, and in California, the City of Burbank power plant has used tertiary municipal effluent since 1967 (Crook, et. al., 1993 and EPA, 1992).

The use of reclaimed water for industrial process water has been less extensive. The acceptability of reclaimed water for this use is dependent on the specific application and is highly variable. In some instances, relatively low quality secondary effluent may be used for applications such as the manufacture of concrete (Crook, et. al., 1993). However, higher quality water is required for other industrial applications, e.g., textile or paper mill process water (EPA, 1992). Two paper mills use tertiary treated effluent from the Los Angeles County Sanitation Districts' Pomona Water Reclamation Plant as process water. The Garden State Paper Company uses 3 mgd of reclaimed water for newsprint reprocessing, and the Simpson Paper Company uses 1 mgd for the manufacture of high quality paper for stationary and wrappings. The tertiary treatment at the Pomona plant includes carbon adsorption for color removal of the wastewater (Crook, et. al., 1993).

**Water Quality Issues for Industrial Process Water.** The suitability of reclaimed water for use in industrial processes depends upon the intended use. At the National Spinning Company, the reclaimed

wastewater from the City of Washington is intended to be used directly in the application of dyeing yarns. Waters used in the textile industry must be non-staining; hence they must be low in turbidity, color, iron, and manganese. A list of requirements for process water in the textile industry is shown in Table 2.1

At the National Spinning Company, the water is also used in high temperature heat exchangers. Water quality criteria to meet industrial cooling water requirements have been developed. These recommended limits are shown in Table 2.2. These parameters were selected to minimize the effects of scaling, corrosion, fouling and biological growth in these systems (Sundberg, et. al., 1991 and EPA, 1992).

**Table 2.1 - Textile Process Water Quality Requirements**

Parameter	Textile Uses	
	Sizing suspension	Scouring, bleach & dye
Cu, mg/l	0.01	
Fe, mg/l	0.3	0.1
Mn, mg/l	0.05	0.01
Hardness, mg/l	25	25
TDS, mg/l	100	100
TSS, mg/l	5	5
Color	5	5

Source: Water Pollution Control Federation, 1989.

Table 2.2. Recommended Cooling Water Quality Requirements

Parameter <sup>a</sup>	Recommended Limit <sup>b</sup>
Cl	500
TDS	500
Hardness	650
Alkalinity	350
pH <sup>c</sup>	6.9-9.0
COD	75
TSS	100
Turbidity <sup>c</sup>	50
BOD <sup>c</sup>	25
Organics <sup>d</sup>	1.0
NH <sub>4</sub> -N <sup>c</sup>	1.0
PO <sub>4</sub> <sup>c</sup>	4
SiO <sub>2</sub>	50
Al	0.1
Fe	0.5
Mn	0.5
Ca	50
Mg	0.5
HCO <sub>3</sub>	24
SO <sub>4</sub>	200

<sup>a</sup> All values in mg/l except pH.

<sup>b</sup> Water Pollution Control Federation, 1989.

<sup>c</sup> From Goldstein, et. al., 1979.

<sup>d</sup> Methylene blue active substances



**Federal and State Regulations.** At present, the process water used by National Spinning Company (a groundwater source) is not used for potable water and is not regulated. However, conversion of this process water to reclaimed wastewater will undoubtedly require National Spinning Company to meet some minimal standards to protect employee health.

Currently, no federal regulations exist governing water reclamation and reuse in the United States (Crook, et. al., 1993). Many states, however, have adopted some form of water reclamation and reuse regulations or guidelines. In a number of states these regulations vary with the intended reclaimed wastewater usage. The variation in regulations for the State of California are shown in Table 2.3 (Crook, et. al., 1993).

Only six states have adopted restrictions directed at industrial use of reclaimed water. These regulations are displayed in Table 2.4. In North Carolina few regulations have been developed that address wastewater reclamation. Regulations developed in 1993 (Crook, et. al., 1993) contain some requirements for land application of domestic wastewater on golf courses and other public access areas. However, no state regulations presently exist that would govern the use of reclaimed wastewater proposed at the National Spinning Company. The application at National Spinning Company would be a unique situation. The North Carolina Division of Environmental Management has stated their willingness to review reuse applications other than those listed in the present state guidelines on a case-by-case basis (Sullins, 1993).

Table 2.3. California Treatment and Quality Criteria for Reuse

<u>Type of Use</u>	<u>Total Coliform</u>	
	<u>Limits</u>	<u>Treatment Required</u>
Fodder, Fiber, and Seed Crops Surface Irrigation of Orchards and Vineyards	---	Primary
Pasture for Milking Animals Landscape Impoundments Landscape Irrigation (Golf Courses, Cemeteries, etc.)	23/100 ml	Oxidation & Disinfection
Surface Irrigation of Food Crops Restricted Recreational Impoundments	2.2/100 ml	Oxidation & Disinfection
Spray Irrigation of Food Crops Landscape Irrigation (Parks, Playgrounds, etc.)	2.2/100 ml	Oxidation, Coagulation, Clarification, Filtration, & Disinfection
Nonrestricted Recreational Impoundments		
Groundwater Recharge	Case-by-Case Evaluation	Case-by-Case Evaluation

Source: Crook, et. al., 1993

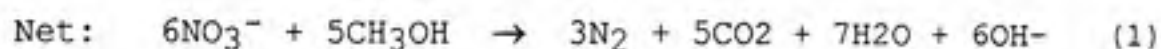
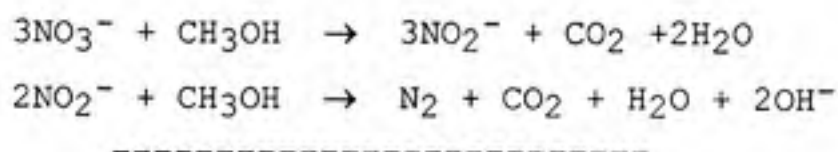
Table 2.4. - State Standards for Industrial Reuse

	Arizona	Hawaii	Nevada	Oregon	Texas	Utah
Reclaimed Water Quality and Treatment Requirements	<ul style="list-style-type: none"> <li>• Determined on a case-by-case basis</li> </ul>	<ul style="list-style-type: none"> <li>• Oxidized and disinfected</li> <li>• Total coliform &lt; 23/100ml (7-day mean) &lt; 240/100ml (2 consecutive samples)</li> </ul>	<ul style="list-style-type: none"> <li>• Secondary treatment with disinfection</li> <li>• Fecal coliform &lt; 200/100ml (mean) &lt; 400/100ml (single sample)</li> </ul>	<ul style="list-style-type: none"> <li>• Biological treatment and disinfection</li> <li>• Total coliform &lt; 240/100ml (2 consecutive samples), &lt; 23/100ml (median)</li> </ul>	<ul style="list-style-type: none"> <li>• 30 mg/L BOD with treatment using pond system</li> <li>• 20 mg/L BOD with treatment other than pond system</li> <li>• Fecal coliform not to exceed 200/100ml</li> </ul>	<ul style="list-style-type: none"> <li>• Advanced treatment</li> <li>• BOD &lt; 10 mg/L at any time</li> <li>• TSS &lt; 5 mg/L at any time</li> <li>• Total coliforms &lt; 3/100ml at any time</li> </ul>

Source: EPA/625/R-92/004 "Manual-Guidelines for Water Reuse", 1992.

## Denitrification

The City of Washington is planning to upgrade their wastewater treatment plant to include a denitrification filter for the removal of nitrogen from the effluent. In the influent to biological wastewater treatment systems, nitrogen is commonly found in the form of ammonium and organic nitrogen. In the treatment process, extended aeration systems can convert ammonium ( $\text{NH}_4^+$ ) to the more oxidized states of nitrite ( $\text{NO}_2$ ) and nitrate ( $\text{NO}_3$ ). Under oxygen-deficient conditions, a number of facultative bacteria, including *Pseudomonas* and *Bacillus*, are able to convert oxidized forms of nitrogen to elemental nitrogen gas ( $\text{N}_2$ ). In order for the microorganisms to reduce the nitrogen compounds, an exogenous carbon supply must be provided. Typically, the source of carbon used for this purpose is methanol ( $\text{CH}_3\text{OH}$ ) (Savage, 1985). The biologically mediated process of converting nitrate to nitrogen gas is termed denitrification. Methanol serves as the electron donor and nitrate as the electron acceptor in the biological oxidation process. The stoichiometry of the overall reaction can be shown as (Jeris, et. al., 1975):



Nitrogen gas is ultimately released to the atmosphere.

In this reaction, five moles of methanol are required to reduce six moles of nitrate-nitrogen to elemental nitrogen. This stoichiometric ratio is equivalent to 1.9 mg of methanol for each mg of nitrate-nitrogen. McCarty, et. al. (1969) found that an additional 30% of methanol was needed to satisfy the growth requirements of the microbes such that:

$$C_m = 2.47 (\text{NO}_3\text{-N}) + 1.53 (\text{NO}_2\text{-N}) + 0.87 (\text{DO}) \quad (2)$$

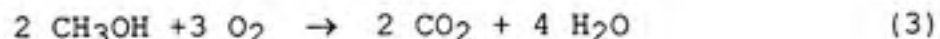
where,

- $C_m$  = required methanol concentration, mg/l
- $\text{NO}_3\text{-N}$  = influent nitrate-nitrogen concentration, mg/l
- $\text{NO}_2\text{-N}$  = influent nitrite-nitrogen concentration, mg/l
- $\text{DO}$  = influent dissolved oxygen concentration, mg/l

Others have verified this equation experimentally as well (Smith, et. al., 1972; Jeris, et. al., 1975; Upton, et. al.; 1993).

The addition of methanol to the influent of the denitrification process will impose a chemical oxygen demand (COD) as well as show an increase in dissolved organic carbon (DOC). The theoretical relationships between these constituents for methanol are as follows:

COD:



or 3 moles oxygen demand for every 2 moles of methanol. This relates in terms of measurable concentrations as a ratio of 3:2, COD:methanol.

DOC:

The theoretical carbon to methanol relationship is 12:32 or simply, 1:2.67.

The theoretical relationship, therefore, for COD:DOC, for methanol addition is 4:1, COD:DOC.

**Denitrification Filter Operation.** Biological denitrification can be performed in several reactor configurations, all of which are operated under anoxic conditions. The City of Washington plans to use a down-flow, attached growth system provided by Tetra Technologies, Inc. (Black & Veatch, 1992). In the Tetra Technologies Inc. system, a small gravel medium is provided to allow for microorganism attachment. As the nitrified wastewater, which is enriched with methanol, passes over the stationary media, the suspended solids are removed by filtration. Concurrently, the attached growth of denitrifiers converts oxidized nitrogen to nitrogen gas. The nitrogen gas gradually builds up in the filter which causes headloss to increase (Savage, 1973). Eventually, as

the headloss reaches its maximum level, the filter requires a 'bumping' operation to release the entrained nitrogen gas. Bumping is accomplished by taking the filter out of service and pumping backwash water to the base of the filter at a significant rate to fluidize the media, thus releasing the gas. The bumping process takes about one minute.

The Tetra system has been installed successfully at a number of wastewater plants (Upton, et. al., 1993). At the Hooker's Point Wastewater Treatment Plant in Tampa, Florida, this process has been in operation since the mid 1970's. At the Altamonte Springs Advanced Wastewater Plant in Altamonte Springs, Florida, a similar system has been in operation since the mid 1980's.

## CHAPTER THREE

## DESCRIPTION OF PILOT-PLANT FACILITIES

Description of Pilot-Plant

A pilot-plant was constructed at the Wastewater Research Center of the University of North Carolina in Chapel Hill. The effluent from the existing wastewater treatment facilities at the City of Washington served as the feed water. A large grab sample (55 gal) was collected from the outlet of the final clarifier (before chlorination) and delivered to Chapel Hill on the day of each run.

A schematic of the treatment units used in the pilot-plant tests is given in Figure 3-1. The pilot-plant was operated in a continuous-flow mode for approximately 23 h in each run. The proposed additional treatment units in the conceptual plan for the City of Washington are: denitrification-chlorination-dechlorination-reaeration. It is proposed that the effluent then undergo storage (15 h) before being passed through greensand-ion exchange and a cartridge filter at the National Spinning Company, Inc.

A summary of design parameters pertinent to City of Washington treatment is given in Table 3.1.



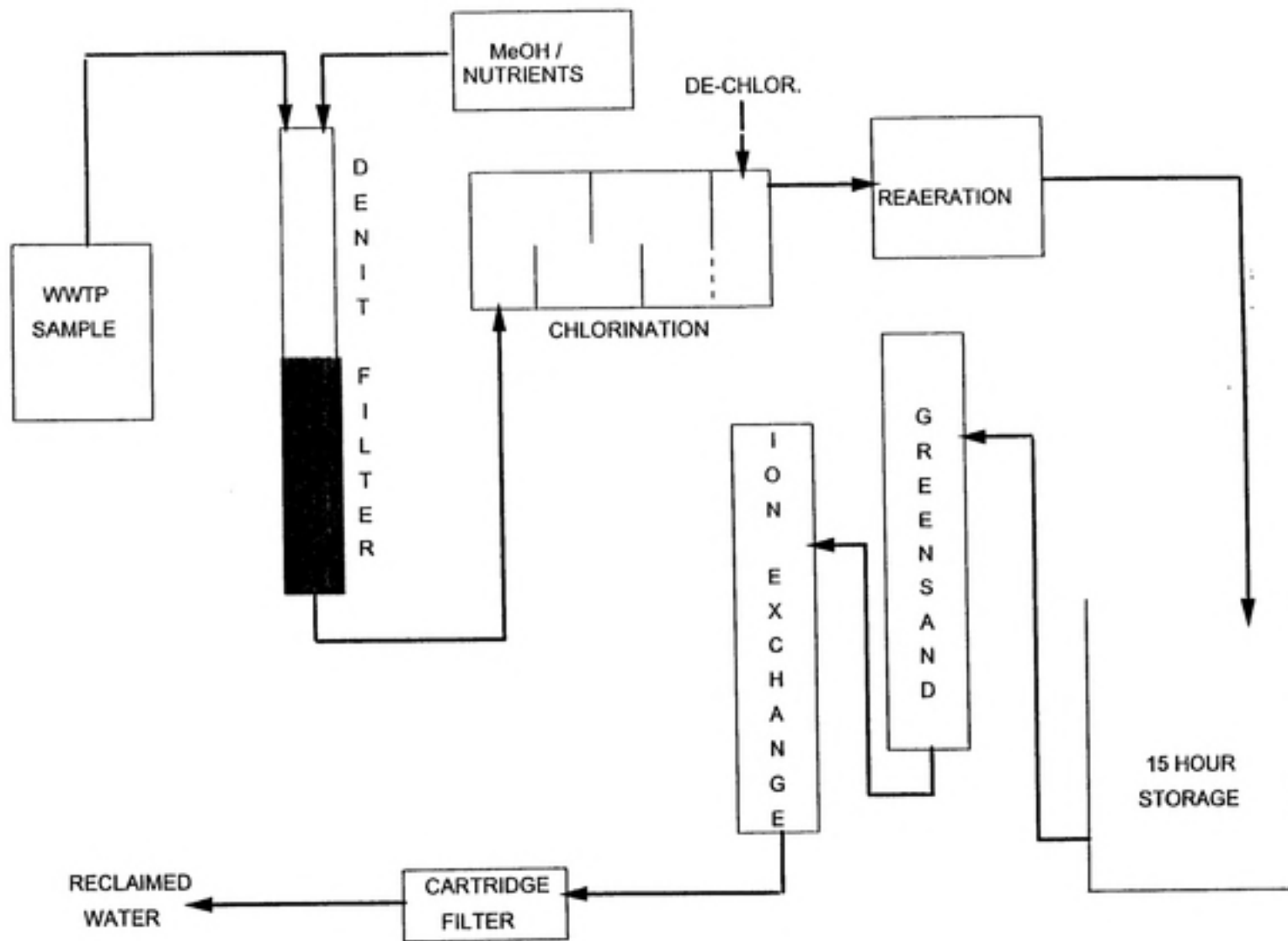


Figure 3-1. Schematic of Pilot-Plant Facilities

Table 3.1 City of Washington Design Parameters

Denitrification	
Empty Bed Contact Time (EBCT)	20 min
Application Rate	2 gpm/sq ft
Influent NO <sub>3</sub> -N	10 mg/l
Carbon Source	Methanol

Chlorination	
Hydraulic Retention Time	31 min
Effluent Total Chlorine	1 mg/l as Cl <sub>2</sub>

Dechlorination	
Effluent Total Chlorine	0 mg/l as Cl <sub>2</sub>

Aeration	
Hydraulic Retention Time	28 min

Storage	
Hydraulic Retention Time	15 h

Source: Black & Veatch, Design Memorandum, 1992.

### City of Washington Treatment

**Denitrification Filter.** A schematic of the Tetra Technologies Inc. filter is provided in Figure 3-2. The filter was designed to achieve the same empty bed contact time (EBCT) and application rate as the full-scale plant. To achieve these design criteria, a reactor was constructed with a diameter of 1-7/8 in and a media depth of 64 in. Filter media was provided by Tetra Technologies, Inc.; a six-inch layer of gravel was used to support the media. The filter column was constructed of Plexiglas and sampling ports were installed at 18 in intervals down the length of the column.

The column was initially seeded with organisms from the Orange Water and Sewer Authority (OWASA) wastewater plant return sludge. A viable population of denitrifiers was achieved within ten days of seeding. Prior to the first experimental run using City of Washington wastewater, the microorganisms were maintained by feeding OWASA final effluent water. After completion of the first experimental run, the denitrifiers were maintained by continuously feeding dechlorinated tap water and nutrients. A cartridge-type filter containing activated carbon (Fulflow, model No. WT-6) served to dechlorinate the tap water. Potassium nitrate was provided for the nitrogen source and Methanol was used as a carbon source. The denitrification filter was maintained with dechlorinated tap water between each of the five experimental runs.

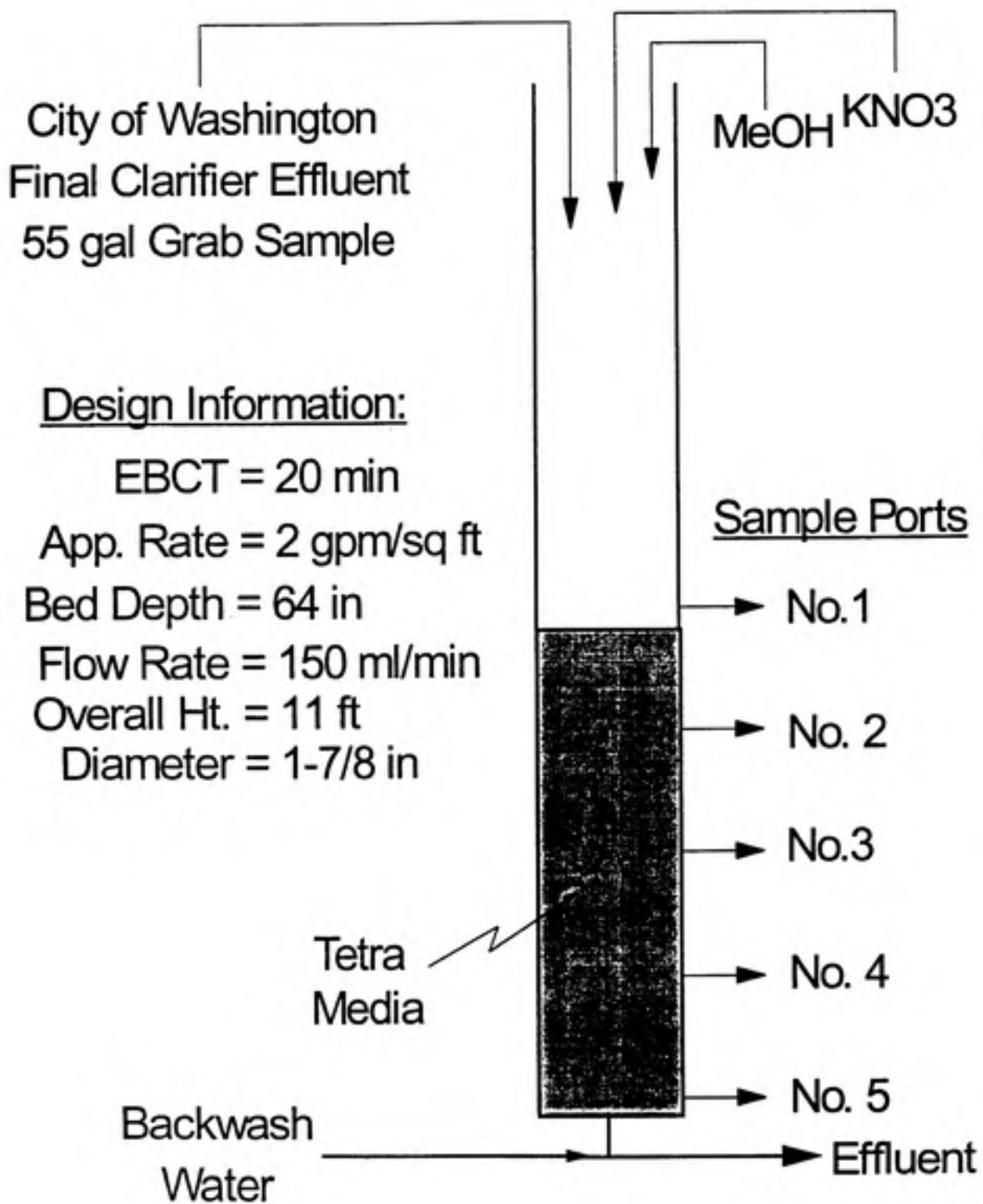


Figure 3-2. Schematic of Tetra Technologies, Inc. Denitrification Filter

During denitrification, nitrogen gas ( $N_2$ ) is produced as nitrate is reduced. Accumulation of  $N_2$  within the media caused significant headloss across the filter. Headloss was allowed to increase in the filter column until such time as necessary to degas the filter by backwashing; this degassing process is referred to as "bumping". Bumping took place every 12 h by pumping (Dayton pump, model no. 6K581A) treated water from the storage reservoir in the upflow direction through the filter for approximately 20 s.

**Chlorination/Dechlorination.** Following denitrification, the effluent was chlorinated and then dechlorinated. The chlorine contact chamber was designed to achieve a hydraulic detention time of 31 min and a residual total chlorine concentration of 1 mg/l as  $Cl_2$ . Approximately 10 mg/l as  $Cl_2$  was added to the influent to achieve this chlorine residual. A chlorine solution (750 mg/l as  $Cl_2$ ) was prepared using household bleach (Clorox). This solution was pumped (Ismatik pump, model no. 7610-20) at a continuous rate of 2 ml/min into the chlorination reactor to achieve 10 mg/l.

Dechlorination was accomplished in a separate reactor downstream of the chlorination reactor. A stock solution of sodium thiosulfate was pumped (Ismatik pump, model no. 7610-20) into the reactor to reduce the chlorine concentration to analytical zero (as  $Cl_2$ ).

**Aeration.** Following dechlorination, the wastewater was aerated. A diffuser stone was used to bubble air into a

reactor that was stirred continuously by a stir bar. A hydraulic detention time of 28 min provided a dissolved oxygen concentration of approximately 7 mg/l.

**Storage.** Following dechlorination and aeration, the water was stored for 15 h in a 50 gal (190 l) plastic tank. After storage, the water was pumped (Masterflex model No. 7553-20 pump and Masterflex model No. 7017-20 pump head ) to the simulated National Spinning Company treatment processes.

#### National Spinning Company Treatment

**Greensand Column.** A greensand filter is used by National Spinning to remove insoluble iron and manganese. In the pilot-plant, the filter was constructed of a plastic, 1.75 in diameter column packed with 24 in of greensand. Greensand media was provided by the National Spinning Company and was of the same type used in their process. This system was operated at a continuous flowrate of approximately 100 ml/min which gave an EBCT of 9.5 min.

**Ion Exchange Column.** Ion exchange filters are used by National Spinning to remove hardness from the water supply. The ion exchange process was simulated in the pilot-plant using a plastic column of the same type used for the greensand filter. Ion exchange media was provided by the National Spinning Company. Effluent from the greensand filter flowed by gravity to the influent of the ion exchange filter. This system was also operated at a continuous flow of approximately 100 ml/min and a bed depth of 24 in which gave an EBCT of 9.5 min.

**Cartridge Filter.** A 5-micron cartridge filter is used by the National Spinning Company downstream of the ion exchange filter to remove any residual suspended matter from the process water. A Cuno filter housing, model No. 1B1, and 5-micron element were provided by the National Spinning Company for use in the pilot process.

#### Description of Pilot-Plant Runs

A total of five batches of the City of Washington wastewater were processed through the pilot-plant. Data for each pilot-plant run, therefore, were obtained from a single batch (grab sample) of wastewater. The complete list of parameters measured in one or more of these runs is provided in Table 3.2. The specific parameters measured in each run are

Table 3.2. Parameters Analyzed in Pilot-Plant

<u>Constituent</u>	<u>Measured Parameter</u>
Particulates	Total Suspended Solids Turbidity Scanning Electron Microscopy
Biodegradable Organics	5-day, Biochemical Oxygen Demand Chemical Oxygen Demand Total Organic Carbon
Volatile Organics	Those listed in SDWA <sup>1</sup> for compliance monitoring
Other Organics	Those listed in SDWA <sup>1</sup> for compliance monitoring
Nutrients	Nitrate-nitrogen
Staining Elements	Iron Manganese National Spinning Fabric Staining Test
Biological Quality	Total Coliforms Fecal Coliforms Heterotrophic Plate Count
Residual Chlorine	Total Chlorine, as Cl <sub>2</sub>
Inorganics	Hardness Sulfite Those listed in SDWA <sup>1</sup> for compliance monitoring
Other	pH Dissolved Oxygen

<sup>1</sup> Safe Drinking Water Act



given in Table 3.3 along with the dates on which each batch (grab sample) was collected.

The treated wastewater received from the City of Washington was amended with potassium nitrate to achieve a concentration of 10 mg/l  $\text{NO}_3\text{-N}$ . This concentration was selected because it represents the worst-case situation expected in actual operation. Methanol (Baxter, industrial grade) was also added to the influent stream as the carbon source (i.e., the electron donor) for the microorganisms. According to the stoichiometry of the denitrification reaction in Equation (1), it is necessary to use a weight ratio of 2.5:1.0 methanol to  $\text{NO}_3\text{-N}$  ratio; thus the concentration of methanol was set at 25 mg/l. Both the nitrate and methanol solutions were pumped (Ismatik pump, model no. 7610-20) into the top of the denitrification filter continuously in the experimental runs. Pump flow rates were measured at regular intervals and adjustments were made to maintain the required concentrations.

After initiation of each run, approximately one hour was needed for the population of microorganisms in the denitrification filter to acclimate to the new source (the filter was fed prior to each experimental run with dechlorinated tap water amended with nitrate and methanol).

Table 3.3. Summary of Pilot-Plant Runs

## Run No. 1, April 21, 1993

- Nitrate-nitrogen
- pH
- Total Chlorine
- Total Suspended Solids
- Chemical Oxygen Demand
- Volatile Organic Chemicals
- Dissolved Oxygen

## Run No. 2, May 21, 1993

- Nitrate-nitrogen
- pH
- Total Chlorine
- Total Suspended Solids
- Chemical Oxygen Demand
- 5-day, Biochemical Oxygen Demand
- Volatile Organic Chemicals
- Dissolved Oxygen
- Iron
- Manganese
- Hardness
- Sulfite

## Run No. 3, July 7, 1993

- Nitrate-nitrogen
- pH
- Total Chlorine
- Total Suspended Solids
- Chemical Oxygen Demand
- 5-day, Biochemical Oxygen Demand
- Volatile Organic Chemicals
- Turbidity
- Dissolved Oxygen
- Iron
- Manganese
- Hardness
- Sulfite

## Batch No. 4, July 30, 1993

- Nitrate-nitrogen
- pH
- Total Chlorine
- SDWA<sup>1</sup> Parameters
- Biological parameters
- Iron
- Manganese
- Total Organic Carbon

## Batch No. 5, November 7, 1993

- Turbidity
- Total Organic Carbon
- Staining Tests
- Scanning Electron Microscopy
- Coagulation Studies

1 Safe Drinking Water Act

The microorganisms were considered acclimated when the nitrate-nitrogen concentration in the denitrification filter effluent was less than about 2 mg/l. Adjustments in the chlorine and sodium thiosulfate dosings were required to achieve a steady chlorine residual in the chlorination effluent and no measurable chlorine in the dechlorination effluent. These adjustments usually required from 3-5 hours.

Upon achieving a dechlorinated effluent, the wastewater was routed to the aeration tank and then to the storage reservoir. After 15 h of continuous storage, the stored water was pumped to the inlet of the simulated National Spinning Company treatment process.

## CHAPTER FOUR

### EXPERIMENTAL METHODS

#### Particulates

**Total Suspended Solids.** Suspended solids were measured in accordance with Standard Method 2540 D (APHA, 1992). Samples were passed through a Whatman Filter GF/F with a particle retention size of 0.7 $\mu$ m. Standard Method 2540 D describes a filter with a particle retention of 1.2 $\mu$ m. Samples were dried in a Fisher, Isotemp Oven, and weighed using a Mettler Instrument Corporation balance, Type H15.

**Turbidity.** Turbidity measurements were performed using a Hach Ratio Turbidimeter, model No. 18900-00. The turbidimeter was calibrated prior to each set of samples analyzed.

**Scanning Electron Microscopy.** Scanning Electron Microscopy (SEM) was performed by Vicki Madden at the University of North Carolina Pathology Department. The procedure involved passing 10 ml of the sample water through a Poretics, 0.2-micron unipore filter. The filter was then mounted on aluminum stubs with colloidal silver paste and coated with Au/Pd alloy.

### Biodegradable Organics

**5-Day Biochemical Oxygen Demand (BOD).** BOD tests were performed in accordance with Standard Method 5210 B (APHA, 1992). A Hach Nutrient Buffer solution, catalog No. 14160-66 and a Hach Nitrification Inhibitor, catalog No. 2533-35 were used for each test. Dissolved oxygen measurements were obtained using the procedure mentioned above.

**Chemical Oxygen Demand (COD).** COD measurements were performed using a Hach COD Reactor, model 45600, in accordance with Standard Method 5220 D (APHA, 1992). Samples were preserved with concentrated sulfuric acid and refrigerated prior to analysis.

**Total Organic Carbon (TOC).** TOC analyses were performed in accordance with Standard Method 5310 (APHA, 1992). An OI Corporation Model No. 700 TOC Analyzer was used for each measurement. Samples were preserved with concentrated sulfuric acid and refrigerated prior to analysis.

**Dissolved Organic Carbon (DOC)** DOC analyses were performed as described above, except that the sample was initially filtered using a Hach 0.45 $\mu$ m glass fiber filter.

### Other Organics.

**Methanol.** Methanol measurements were performed by Oxford Laboratories, Inc. located in Wilmington, North Carolina. Samples were refrigerated and express-mailed to the testing laboratory.

**Safe Drinking Water Act Regulated Chemicals.** The list of chemicals required by the Safe Drinking Water Act (SDWA) for compliance monitoring were analyzed by Oxford Laboratories, Inc., in Wilmington, North Carolina. These chemicals included the 55 volatile organic chemicals (VOC's) listed as well as the complete list of organic and inorganic chemicals. All samples were refrigerated immediately after sampling and express-mailed to the testing laboratory.

### Nutrients

**Nitrate-nitrogen.** Nitrate-nitrogen was measured using an Orion Research nitrate probe, model No. 701A/digital ionalyzer in accordance with instructions provided by the supplier. A calibration curve was developed daily during each experimental run.

### Staining Elements

**Total Iron.** Total iron measurements were performed using the Phenanthroline Method, Standard Method 3500-Fe D (APHA, 1992). Photometric determinations were made using a Hitachi, U-2000 spectrophotometer, at a wavelength of 510 nm. A calibration curve was developed for each test using standard solutions of ferrous ammonium sulfate. Iron tests for batch runs 2 and 3 were performed by Mr. Paul Savard.

**Manganese.** Manganese measurements were performed using the Persulfate Method, Standard Method 3500-Mn D (APHA, 1992). Photometric determinations were made using a Hitachi, U-2000 spectrophotometer, at a wavelength of 525 nm. A calibration curve was developed for each test using standard solutions of potassium permanganate. Manganese tests for batch runs 2 and 3 were performed by Mr. Paul Savard.

**Fabric Staining Analysis.** The test used to determine the acceptance of the reclaimed water from a staining standpoint was developed by National Spinning Company. The procedure was to pass 500 ml of sample water through a Whatman GF/C filter and to visually inspect the filter for evidence of staining. A quantitative value was applied to each sample by utilizing a Zellweger Uster, High Volume Instrument (HVI) at the College of Textiles of North Carolina State University, in Raleigh.

Analyses were performed by Ms. Jan Pegram and involved the measurement of the "whiteness" and "yellowness" of the sample filter. Measured values were then compared using a Cotton Color Grade Chart, a chart used to differentiate grades of cotton.

### Biological Quality

**Microbiological Examinations.** All microbiological tests (Heterotrophic Plate Counts, Total Coliforms and Fecal Coliforms ) were analyzed by outside laboratories. Webb Technical, located in Raleigh, NC, analyzed all of the samples with the exception of one groundwater sample taken from the well supply of the National Spinning Company. This sample was analyzed by the City of Washington.

### Other

**Dissolved Oxygen.** Dissolved oxygen measurements were made using a YSI, model No. 54A oxygen meter in accordance with Standard Method 4500-O G (APHA, 1992). Samples were collected in 300 ml standard BOD bottles and the meter was calibrated prior to each set of analyses.



**Hardness.** Hardness was measured using the EDTA Titrimetric Method, Standard Method 2340 C (APHA, 1992). All measurements were performed by Mr. Paul Savard.

**pH.** The pH was measured using a Fisher Accumet pH meter, model No. 610. Calibration was performed a minimum of once daily during each experimental run.

**Total Chlorine Residual.** Total chlorine measurements were performed using the DPD Ferrous Titrimetric Method, Standard Method 4500-Cl F (APHA, 1992). Both free and combined chlorine were determined using this method.

## CHAPTER FIVE

## EXPERIMENTAL RESULTS

Denitrification Process

The stability of the denitrification process was of primary concern due to the switching of source waters from dechlorinated tap water to City of Washington wastewater. Nitrate concentrations and pH were measured at regular intervals on the influent and effluent of the Tetra denitrification filter. Plots of these results for the first four experimental runs are shown in Figures 5-1 and 5-2. The beginning time shown in each figure corresponds to the start of the wastewater feed from the City of Washington. The results indicate that denitrification reached a steady state very quickly, in most cases within one hour. The denitrification process was stable throughout the pilot tests as indicated by the small variation in  $\text{NO}_3\text{-N}$  (Figure 5-1) and pH (Figure 5-2) in the effluent of the filter. Minor variations in performance could be explained by fluctuations in the influent feed rate; adjustments were made in pumping rate periodically during each test. The data from the final experimental run are not included in Figures 5-1 and 5-2 due to a limited number of samples; however, the results were similar to those obtained in the first four runs.

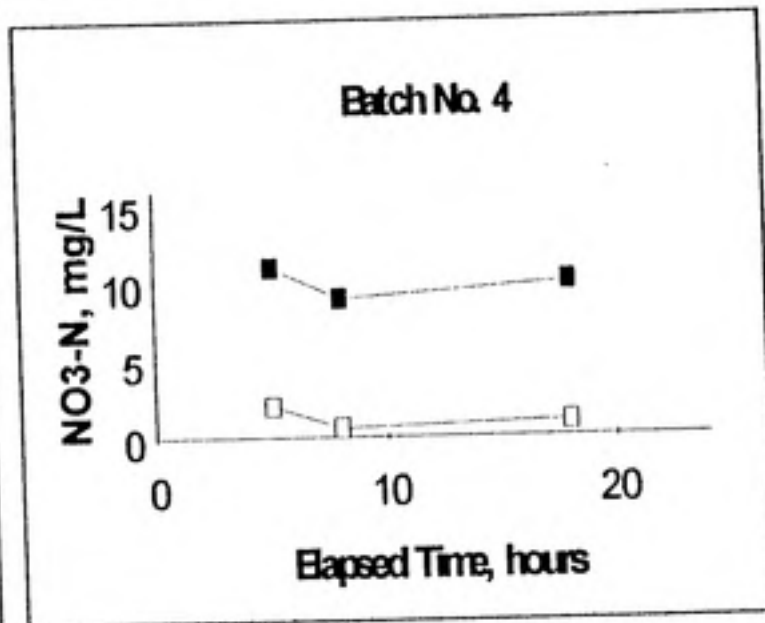
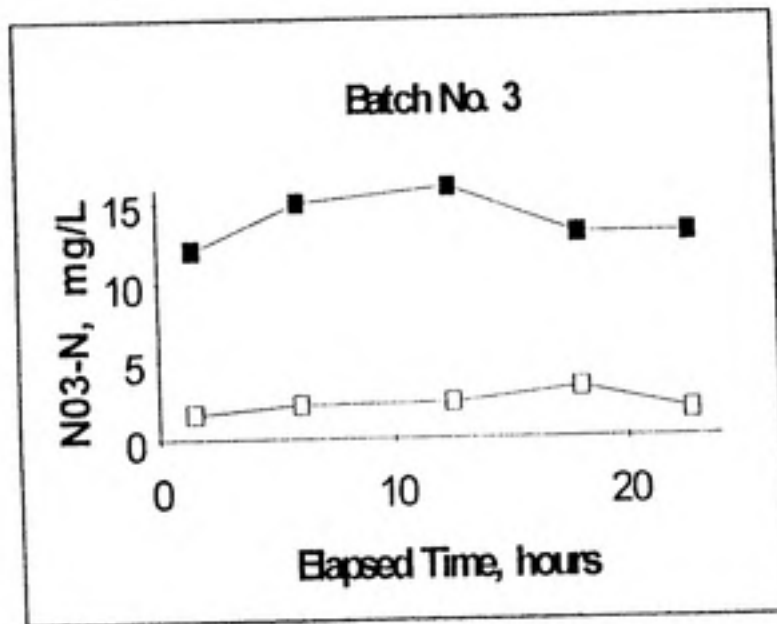
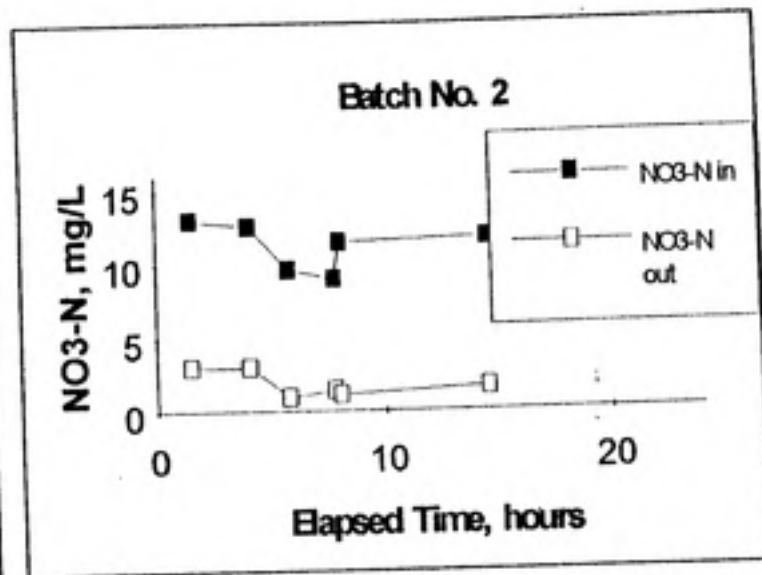
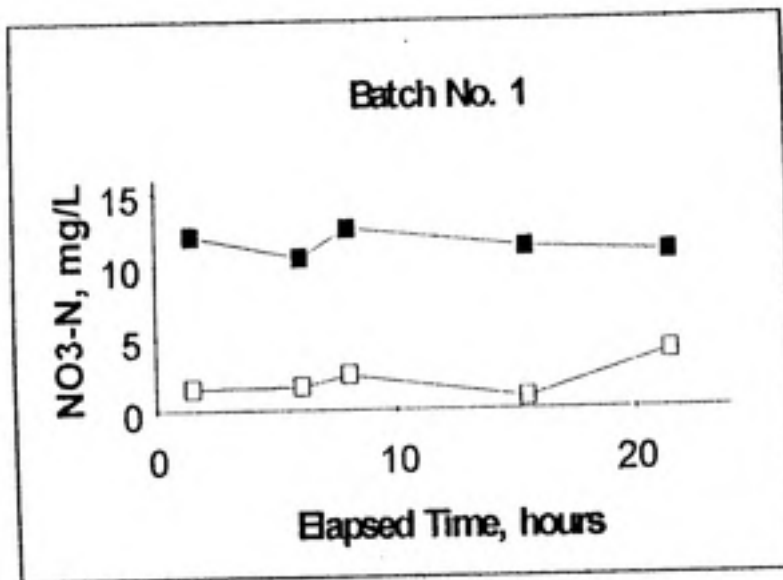


Figure 5-1. Nitrate Variability During Batch Runs 1-4.

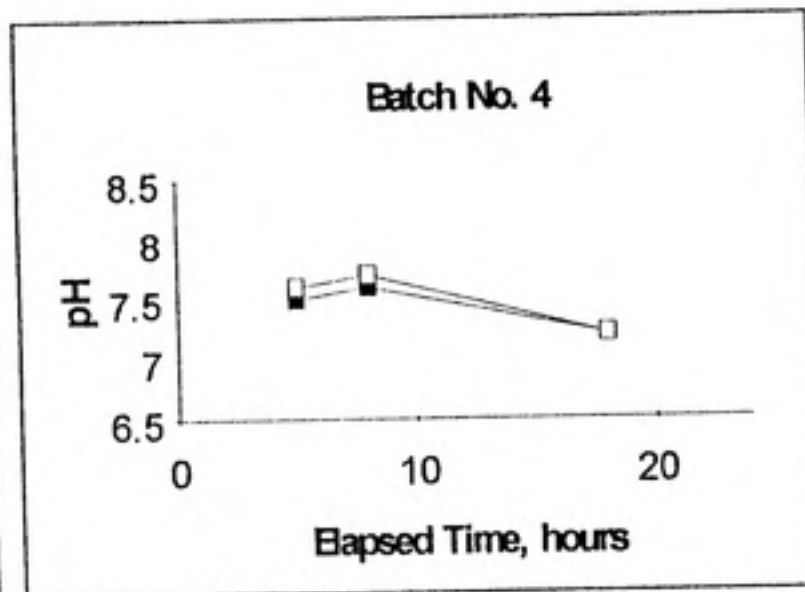
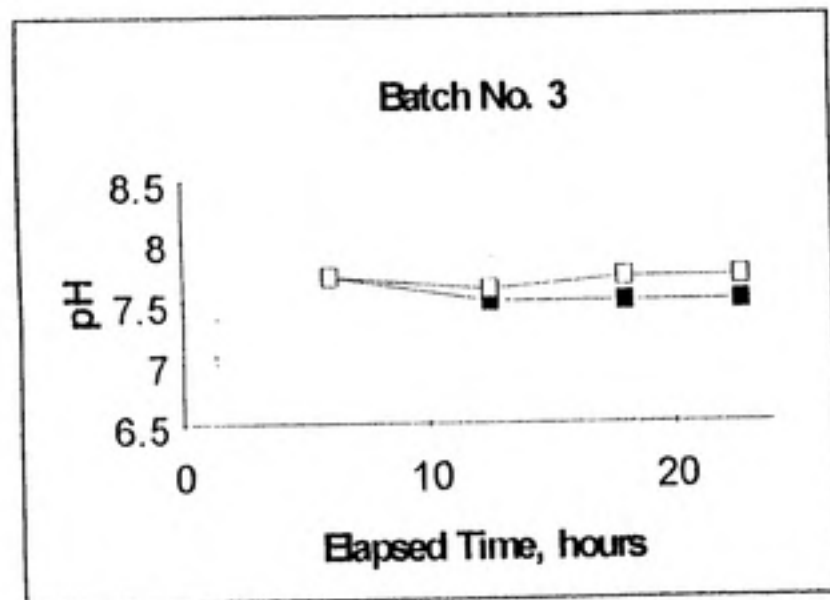
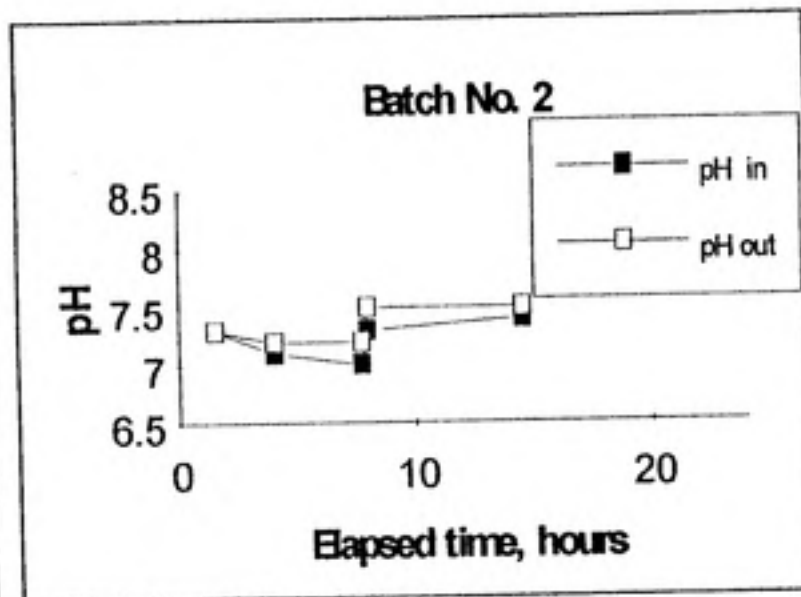
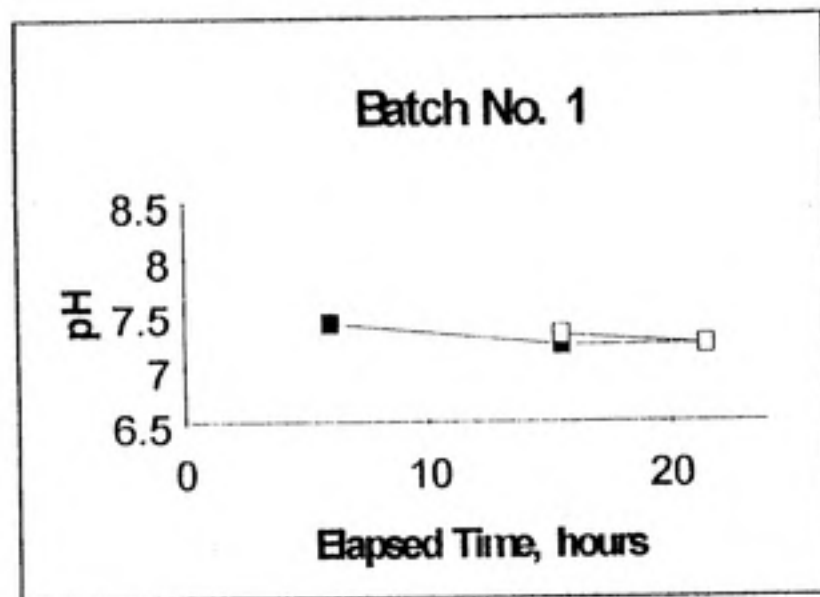


Figure 5-2. pH Variability During Batch Runs 1-4.

Nitrate removal efficiency in the filter averaged approximately 84 percent during the first four experimental runs. To maintain the population of denitrifiers between experimental runs, dechlorinated tap water was fed to the denitrification filter. Measurements of nitrate removal with dechlorinated tap water were taken on the day preceding each experimental run. These results showed a nitrate removal percentage of 90 percent, slightly higher than that achieved during the actual experimental runs. A possible explanation for the lower removal rates with City of Washington wastewater may be that interfering ions may be present in the wastewater that are not present in the tap water. Measurement of nitrate using the Nitrate Electrode Method (Standard Method 4500-NO<sub>3</sub><sup>-</sup> D., 1992) is affected by significant concentrations of chloride and bicarbonate ions in the water sample. Although actual chloride and bicarbonate alkalinity were not measured in the Washington wastewater, it is quite possible that these concentrations had some affect on the measured nitrate concentrations.

The results of measuring various process parameters related to nitrate removal within the filter are shown in Table 5.1. Nearly all of the nitrate was removed in the top 10 in thus showing that the fixed-film system is very effective. Consistent with the denitrification process, dissolved oxygen (DO) concentration decreased sharply with depth in the filter. The decrease in chemical oxygen demand (COD) and dissolved

organic carbon (DOC) concentrations with depth is also expected due to utilization of methanol by the attached microorganisms during denitrification.

Table 5.1. Denitrification Filter Profile

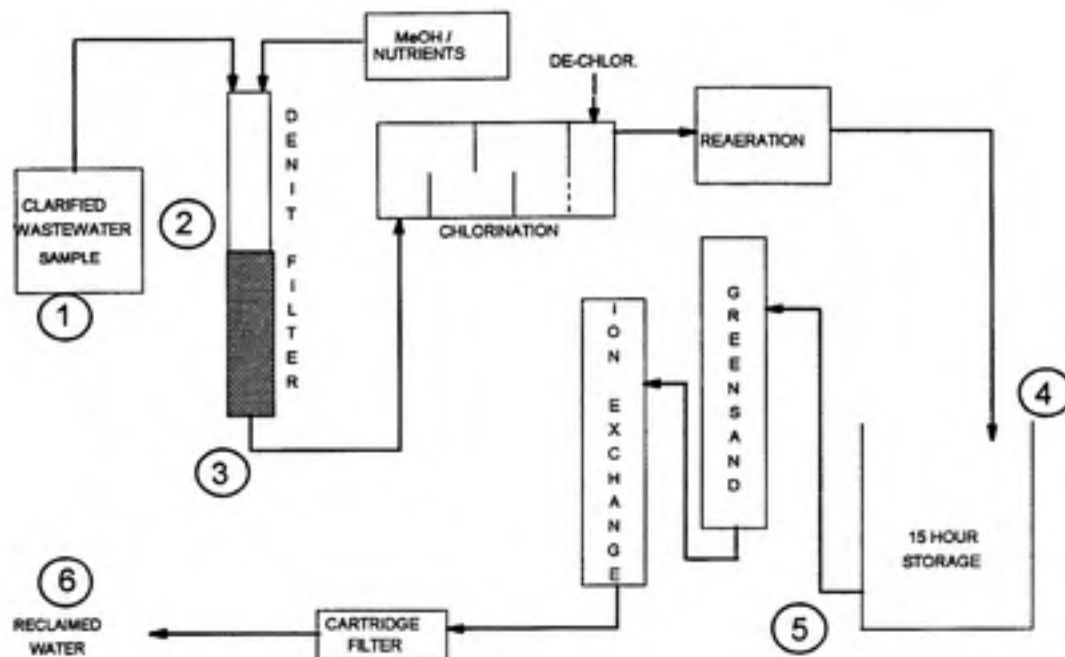
Sample Port	Distance From Top of Media (in)	DO (mg/l)	NO <sub>3</sub> -N, (mg/l)	pH	COD (mg/l)	DOC (mg/l)
1	-8	4.6	8	7.4	54.2	23.0
2	10	0.8	1.3	7.4	17.8	14.7
3	28	1.2	1	7.4	18.8	7.4
4	46	0.8	1	7.4	20.4	8.3
5	64	0.9	1.2	7.3	15	6.5

Theoretically, for each mg/l of methanol removed by the filter, a corresponding reduction in COD of 1.5 mg/l should result. From the results of the denitrification filter operation, 39.2 mg/l of COD was removed during operation, corresponding to the removal of 26.1 mg/l methanol. The amount of methanol added to the filter was approximately 25 mg/l, or a factor of 2.5 times the influent NO<sub>3</sub>-N concentration. This compares very favorably to the measured COD results. A comparison of predicted versus actual COD:DOC can not be made for these data as the DOC samples were filtered to remove any particulates present. The COD samples were not filtered.

### General Indicators of Reclaimed Wastewater Quality

Treatment of the City of Washington wastewater includes dechlorination and reaeration processes in order to meet effluent toxicity and dissolved oxygen discharge limits. These two conditions will encourage microbial activity and thus further changes in quality of wastewater before use by the National Spinning Company. A significant regrowth of microorganisms could result in a deteriorated water quality and fouling of treatment units at National Spinning Company.

**General Parameters.** Indirect measures of water quality changes included DO, COD, five-day biochemical oxygen demand (BOD<sub>5</sub>), and DOC given in Table 5.2. The results listed for DO, COD and BOD<sub>5</sub> in Table 5.2 are average values of grab samples taken during the first three experimental runs; DOC was measured in Run No. 5. The values of COD (10 mg/l), BOD<sub>5</sub> (2.0 mg/l) and DOC (8.5 mg/l) are fairly low, indicating a well-treated wastewater. Very little depletion in DO occurred (from 7.6 to 6.4 mg/l) during the 15 h of storage. The increase in COD and BOD<sub>5</sub> at the Tetra denitrification influent represents the addition of methanol into the system. There is also little change in BOD<sub>5</sub> from the effluent of the Tetra denitrification filter to the end of the storage time. The increase in TOC during storage may be artifactual. The DOC samples were first filtered to remove insoluble materials and some organics may have leached from the filter into the sample.



Sample Location	Description	DO (mg/l)	COD (mg/l)	BOD <sub>5</sub> (mg/l)	DOC (mg/l)
1	City of Washington Secondary Clarifier Effluent	not measured	15.3	0.9	54 <sup>1</sup>
2	Denitrification Filter Influent	4.6	56.1	38.1	23
3	Denitrification Filter Effluent	0.8	20.9	3.3	6.5
4	Pre Storage	7.6 <sup>2</sup>	not measured	not measured	7.1
5	Post Storage	6.4	14.2	2.1	13.2
6	Reclaimed Wastewater	not measured	10.0	2.0	8.5

<sup>1</sup> unfiltered sample, (Total Organic Carbon)

<sup>2</sup> reaeration increased DO

Table 5.2. Results of General Water Quality Parameters



**Bacteriological Examination.** Standard bacteriological indicators were compared for the reclaimed wastewater and the existing process waste used by National Spinning Company (Table 5.3). These results were obtained during experimental Run No. 4.

**Table 5.3. Comparison of Bacteriological Results**

	Fecal Coliforms	Total Coliforms	Heterotrophic Plate Count
National Spinning Company Process Water	< 1/ml	< 2/ml	11/ml
Reclaimed Wastewater	< 1/ml	9/ml	57,000/ml

The fecal and total coliform results are within limits adopted by other states for industrial reuse applications. Although only one sample was measured, it is evident that the reclaimed wastewater is of poorer quality than the existing process water when based on total coliforms and heterotrophic bacteria. Because the wastewater had been chlorinated, the results indicate that regrowth may have occurred during reaeration or the 15 h storage period.

Regrowth of microorganisms should always be expected during the storage of dechlorinated, reaerated wastewater regardless of the extent of treatment. To prevent subsequent problems of fouling in water treatment at the National Spinning

Company, it is suggested that some means of disinfection be applied at the storage reservoirs, either on a continuous or intermittent basis. After treatment with greensand, ion exchange and cartridge filters, however, it will be necessary to add a reducing agent to again eliminate chlorine, if chlorine were used for disinfection, before the yarn dyeing. Chlorine cannot be present because it can react with dyes and give imperfections in colors.

Tests for the presence of viruses and individual pathogens were not conducted as part of this study. Analysis of these organisms would likely be required to determine potential employee and customer health risks. It should be noted that during the National Spinning Company dyeing process, the water is heated to temperatures in excess of 180°F. Although this will not guarantee a sterile water, it will certainly reduce the presence of these microorganisms and consequently reduce the potential health risks.

**Measurement of SDWA Regulated Chemicals.** Process water at the National Spinning Company is used in open vat dyeing operations. Consequently, there is concern of employee exposure to volatile organic chemicals (VOCs) remaining in the reclaimed wastewater. Analyses of 55 VOCs were performed on the wastewater after storage but before treatment in the process water scheme used by National Spinning Company. Grab

samples were taken during Run Nos. 1-3 after the pilot-plant had reached stable operation. The list of 55 VOCs is the same as that done for quarterly compliance monitoring by water utilities in accordance with the Safe Drinking Water Act Amendments (SDWA).

Results of VOC analyses are given in Table 5.4. Results reported in Table 5.4 are average values of one grab sample taken during each of Run Nos. 1-3. The only compounds that were present in concentration exceeding the detection limit of 0.5 parts per billion (ppb) were chloroform (11.4 ppb), bromodichloromethane (5.4 ppb) and chlorodibromomethane (0.9 ppb). These chemicals are expected as a result of chlorination at the City of Washington water treatment plant and in the pilot plant process. The total concentration of these trihalomethanes (17.7 ppb) is well below the current drinking water standard of 100 ppb.

Run No. 4 was conducted to compare the reclaimed wastewater directly with that presently being used for process water at the National Spinning Company. As a means of comparison, both samples were analyzed for the presence of all those contaminants required by the Safe Drinking Water Act for compliance monitoring with the exception of the 55 VOCs. This latter group had been analyzed in previous samples and found (with the exception of THMs) to be absent (see Table 5.4).

**Table 5.4. Analysis of Treated and Stored Wastewater for 55 VOCs Contained on SDWA Compliance List. Reported Values are Averages for Run Nos. 1-3.**

VOC	Concentration ppb	VOC	Concentration ppb
Trihalomethanes		1,1,2-Trichloroethane	< 0.5
Chloroform	11.6	Tetrachloroethylene	< 0.5
Bromodichloromethane	5.54	1,3-Dichloropropane	< 0.5
Chlorodibromomethane	0.85	Chlorobenzene	< 0.5
Bromoform	< 0.5	Ethylbenzene	< 0.5
Compounds	< 0.5	1,1,1,2-Tetrachloroethane	< 0.5
Dichlorodifluoromethane	< 0.5	Total Xylenes	< 0.5
Chloromethane	< 0.5	Styrene	< 0.5
Vinyl Chloride	< 0.5	Isopropylbenzene	< 0.5
Bromomethane	< 0.5	1,1,2,2-Tetrachloroethane	< 0.5
Chlorethane	< 0.5	1,2,3-Trichloropropane	< 0.5
Fluorotrichloromethane	< 0.5	n-Propylbenzene	< 0.5
1,1-Dichloroethylene	< 0.5	Bromobenzene	< 0.5
Dichloromethane	< 0.5	1,3,5-Trimethylbenzene	< 0.5
trans-1,2-Dichloroethylene	< 0.5	o-Chlorotoluene	< 0.5
1,1-Dichloroethane	< 0.5	p-Chlorotoluene	< 0.5
2,2-Dichloropropane	< 0.5	tert-Butylbenzene	< 0.5
cis-1,2-Dichloroethylene	< 0.5	1,2,4-Trimethylbenzene	< 0.5
Bromochloromethane	< 0.5	sec-Butylbenzene	< 0.5
1,1,1-Trichloroethane	< 0.5	p-Isopropyltoluene	< 0.5
1,1-Dichloropropene	< 0.5	1,3-Dichlorobenzene	< 0.5
Benzene	< 0.5	1,4-Dichlorobenzene	< 0.5
1,2-Dichloroethane	< 0.5	n-Butylbenzene	< 0.5
Trichloroethylene	< 0.5	o-Dichlorobenzene	< 0.5
1,2-Dichloropropane	< 0.5	1,2,4-Trichlorobenzene	< 0.5
Dibromomethane	< 0.5	Hexachlorobutadiene	< 0.5
1,3-Dichloropropene	< 0.5	Napthalene	< 0.5
Toluene	< 0.5	1,2,3-Trichlorobenzene	< 0.5

The objective of the SDWA analysis was to compare the two waters and not to determine whether the reclaimed wastewater met criteria as a drinking water source. Oxford Laboratories, in Wilmington, North Carolina, performed the chemical analysis on both waters. Table 5.5 shows the results obtained along with the maximum contaminant level (MCL) for each chemical (Pontius, 1990). All but one of the chemicals is well below the MCL and, in fact, below the detection limit of the analytical method (designated by < ). The sole exceptions are phthalates. This group of chemicals derive from plasticizers which are often found in analysis of wastewater effluents. In addition, contact of water with plastic materials used in the pilot plant could also produce phthalates and thus give an artifactual result.

Table 5.5. Comparative Analysis of Chemicals on SDWA Compliance Monitoring List for Existing Process Water at National Spinning Company and for Reclaimed wastewater.

Contaminant	MCL (mg/l)	Reclaimed Water (mg/l)	NSC Process Water (mg/l)	Contaminant	MCL (mg/l)	Reclaimed Water (mg/l)	NSC Process Water (mg/l)
<b>Organics</b>				Pentachlorophenol	0.001	<0.00004	<0.00004
Adipates	0.5	<0.0006	<0.0006	Phthalates	0.004	0.15	<0.0006
Alachlor	0.002	<0.0002	<0.0002	Picloram	0.5	<0.0001	<0.0001
Aldicarb	0.003	<0.0005	<0.0005	Propachlor		<0.00005	<0.00005
Aldicarb Sulfone	0.002	<0.0008	<0.0008	PCB's	0.0005	<0.0001	<0.0001
Aldicarb sulfoxide	0.004	<0.0005	<0.0005	Simazine	0.004	<0.00007	<0.00007
Aldrin		<0.00002	<0.00002	Toxaphene	0.005	<0.001	<0.001
Atrazine	0.003	0.00014	<0.0001	2,4,5-TP	0.05	<0.0002	<0.0002
Benzo(a)pyrene		<0.00002	<0.00002	<b>Inorganics</b>			
Butachlor		<0.00015	<0.00015	Antimony	0.01/0.005	<0.001	<0.001
Carbofuran	0.04	<0.0008	<0.0008	Arsenic	0.05	<0.005	<0.005
Chlordane	0.002	<0.0002	<0.0002	Asbestos	7 MFL		
2,4-D	0.07	<0.0001	<0.0001	Barium	2	<0.020	0.085
Dalapon	0.2	<0.001	<0.001	Beryllium	0.001	<0.001	<0.001
Dibromochloropropane	0.0002	<0.00002	<0.00002	Cadmium	0.005	<0.002	<0.002
Dieldrin		<0.00001	<0.00001	Chromium	0.1	<0.005	<0.005
Dicamba		<0.0001	<0.0001	Copper	1.3	0.006	<0.003
Dinoseb	0.007	<0.0001	<0.0001	Cyanide	0.2	<0.005	<0.005
Diquat	0.02	<0.0004	<0.0004	Fluoride	4	0.63	1.08
Endothall	TT.1	<0.009	<0.009	Iron		0.500	0.015
Endrin	0.002	<0.00001	<0.00001	Lead	-	<0.003	<0.003
Ethylene dibromide	0.00005	<0.00001	<0.00001	Manganese		0.055	0.020
Glyphosate	0.7	<0.006	<0.006	Mercury	0.002	<0.0002	<0.0002
Heptachlor	0.0004	<0.00004	<0.00004	Nickel	0.1	<0.005	<0.005
Heptachlor epoxide	0.0002	<0.00002	<0.00002	Nitrate, as N	10	1.08	<0.1
Hexachlorobenzene	0.001	<0.0001	<0.0001	Nitrite, as N	1	<0.1	<0.1
Hexachlorocyclopentadiene	0.05	<0.0001	<0.0001	Nitrate + Nitrite, as N	10	1.08	<0.2
3-Hydroxycarbofuran		<0.0006	<0.0006	Selenium	0.05	<0.005	<0.005
Lindane	0.0002	<0.00002	<0.00002	Sulfate	400/500	50	46
Methomyl		<0.0005	<0.0005	Thallium	0.002/0.001	<0.001	<0.001
Methoxychlor	0.04	<0.0001	<0.0001	pH (lab)		7.6	8.85
Metolachlor		<0.00025	<0.00025				
Metribuzin		<0.0002	<0.0002				
Oxamyl	0.2	<0.002	<0.002				

### Process Water Quality Requirements

**Iron and Manganese.** Process water at National Spinning Company is used directly in the application of yarn dyeing. The process water, therefore, must not contain significant concentrations of any element that might result in staining of the yarns.

Measurements of iron and manganese were taken after storage of the treated wastewater. These values are compared with levels in the existing National Spinning Company groundwater supply in Table 5.6. The highest iron measurement recorded for the treated wastewater (0.16 mg/l) is very low. The values of iron and manganese obtained in the pilot-plant process were determined to be acceptable by National Spinning Company standards for these elements.

**Table 5.6. Comparison of Iron and Manganese in Existing National Company Raw (Groundwater) Water and Treated Wastewater After 15 h Storage.**

	Iron (mg/l)	Manganese (mg/l)
National Spinning Company Groundwater, Before Treatment	0.10	0.07
Treated Washington Wastewater, Before National Spinning Company Treatment		
Run No. 1	0.16	0.12
Run No. 2	< 0.05	<0.05
Run No. 3	< 0.05	<0.05

**Particulates.** Total suspended solids measurements were made at several locations in the pilot-plant treatment process. A summary of these results is shown in Table 5.7. In each instance, the measurements were less than 5 mg/l, including the City of Washington clarified water sample. This level of suspended solids was deemed acceptable by the National Spinning Company.

**Table 5.7. Removal of Suspended Solids and Turbidity in the Pilot-Plant.**

Sample Description	Turbidity (NTU)	TSS (mg/l)
Washington Secondary Clarifier Effluent	0.87	< 5.0
Tetra Denitrification Filter, Influent	0.92	< 5.0
Tetra Denitrification Filter, Effluent	1.24	< 5.0
Pre Storage	1.25	< 5.0
Post Storage	1.28	< 5.0
Reclaimed Wastewater	1.03	< 5.0
Existing National Spinning Company Process Water	0.18	Not Measured



Measurements of turbidity are also included in Table 5.7. The reclaimed wastewater (1.03 NTU) is considerably more turbid than the present process water (0.18 NTU) used at National Spinning.

**Coagulation Study.** Coagulation of the post-storage water was performed during Run No. 5 to determine the extent of turbidity removal. In this study, a commercially available polymer (River Clear 3103) was added to a sample of the storage effluent water and the water was treated using a conventional jar test treatment scheme: rapid mix, flocculation, and sedimentation. Riverclear polymer was selected based on evaluations performed by the City of Washington in which the effectiveness of different coagulants was compared. Results of the coagulation tests are shown in Figure 5-3. A dosage of 10 mg/l was needed to reduce turbidity to a minimum of 0.18 NTU. This compares closely with the turbidity of the existing National Spinning Company process water ( 0.14 NTU).

**Staining Potential.** One of the tests performed by National Spinning is used to determine the staining potential of the water. Test procedures are outlined in Chapter Five of this report and involve passing 500 ml of water through a 1.2 $\mu$ m glass-fiber filter and analyzing the filter for residual staining. In this test, a considerable amount of staining was present with the reclaimed water as compared to the existing process water.

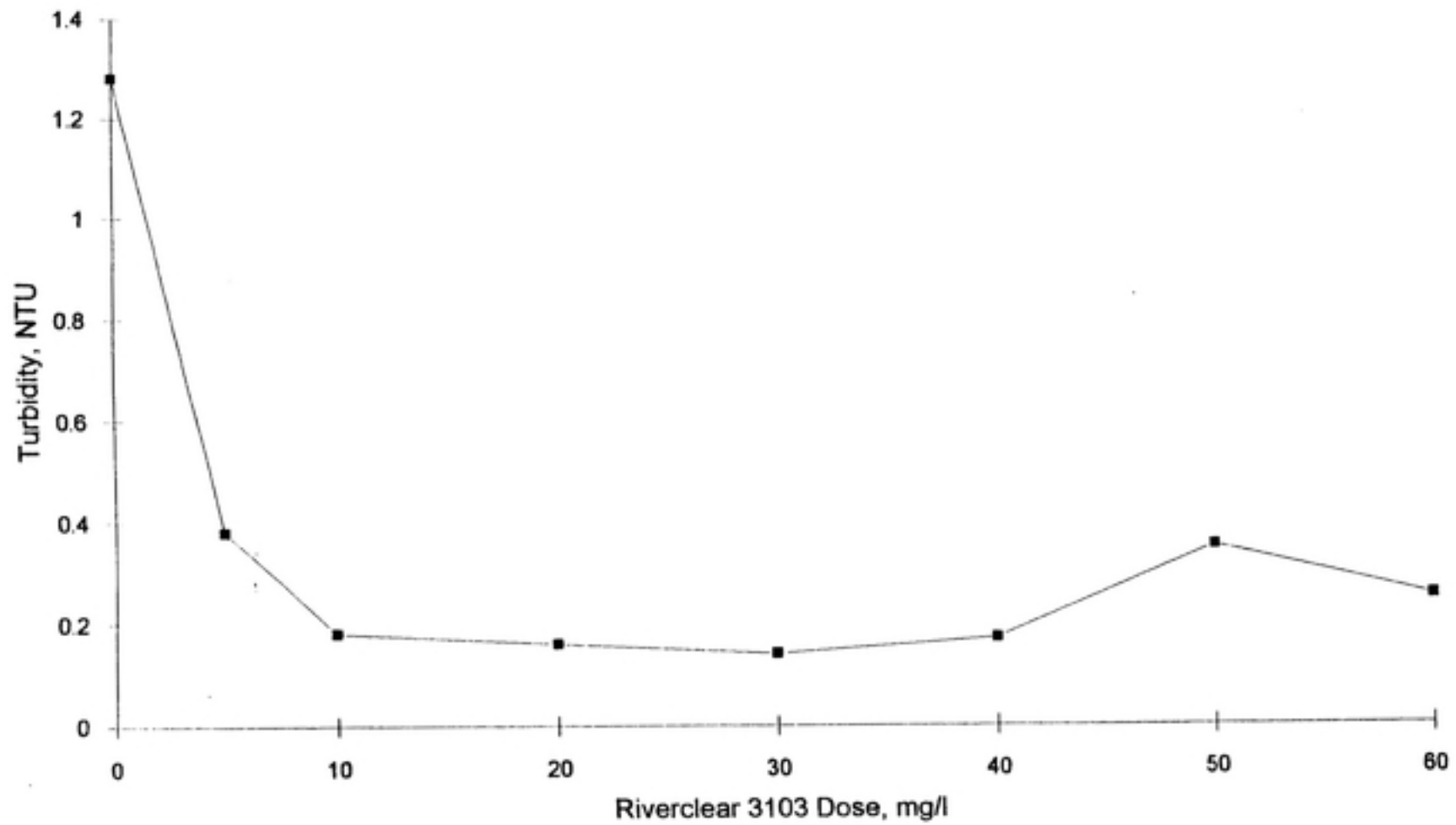


Figure 5-3 Results of Coagulation Study

Photographs of these samples are shown in Figures 5-4a-c. Coagulation of the treated wastewater with Riverclear at a near optimal dosage of 30 mg/l removed much of the residual staining; however, the filter exposed to the coagulated water still showed more staining than that exposed to National Spinning Company process water.

An attempt was made to quantify the amount of staining on each filter. Using a Zellweger Uster, High Volume Instrument (HVI) at the North Carolina State University College of Textiles a comparison was made of each sample using a Cotton Color Grade Chart. These results are displayed in Table 5.8. In this test, higher values indicate more color on the filter, thus a greater potential for fabric staining. The filter sample of the existing process water showed less staining than all of the coagulated water samples. In fact, it was equivalent to a filter sample of Chapel Hill tap water.

**Scanning Electron Microscopy.** Further investigation into the nature of the particles remaining in the reclaimed wastewater was performed using scanning electron microscopy (SEM). This procedure involved passing 10 ml of a sample water through a 0.2  $\mu\text{m}$  unipore filter. The filter was then mounted on aluminum stubs with colloidal silver paste and coated with Au/Pd alloy. The filter was then viewed under an electron microscope. Scanning electron micrographs are shown in Figures 5-5a-c. The reclaimed wastewater contains more particles than the existing process



Figure 5-4a Fabric Staining Potential of Existing National Spinning Company Process Water.

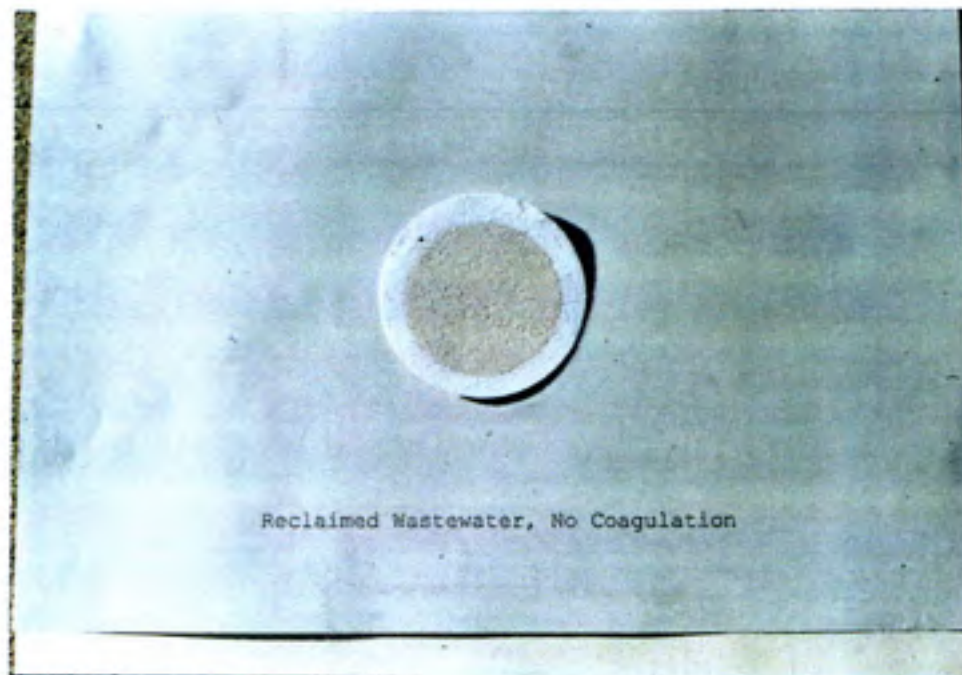


Figure 5-4b Fabric Staining Potential of Reclaimed Wastewater without Coagulation.



Figure 5-4c Fabric Staining Potential of Reclaimed Wastewater with Coagulation (30 mg/l Riverclear 3103).

**Table 5.8. Comparative Analysis of Staining Potential for Existing National Spinning Company Process Water and Treated Wastewater using a Cotton Color Grade Chart.**

Sample Description	Riverclear Dose ( mg/l )	Whiteness	Yellowness
National Spinning Company Process Water <sup>1</sup>	0	5.2	7.8
Reclaimed Wastewater <sup>2</sup>	0	6.9	9.7
Storage Effluent <sup>3</sup>	20	6.0	8.4
Storage Effluent <sup>3</sup>	30	5.8	8.3
Storage Effluent <sup>3</sup>	40	5.7	8.0
Chapel Hill, Tap Water	0	5.3	7.8

<sup>1</sup> after greensand-ion exchange-cartridge filter treatment

<sup>2</sup> after greensand-ion ion exchange treatment

<sup>3</sup> Coagulation performed on storage effluent

water. Coagulation of the treated wastewater does seem to reduce the number of particles significantly; however, not to the level found in the present process water. Higher magnification of a typical particle from the reclaimed wastewater without coagulation is shown in Figure 5-6.

An energy dispersive X-ray analysis was performed on this particle and the results are shown in Figure 5-7. The primary elements found in the particle (iron, manganese, and silica) do not suggest anything atypical about the particle's composition.



Figure 5-5a SEM Sample of Existing Process Water at National Spinning Company.

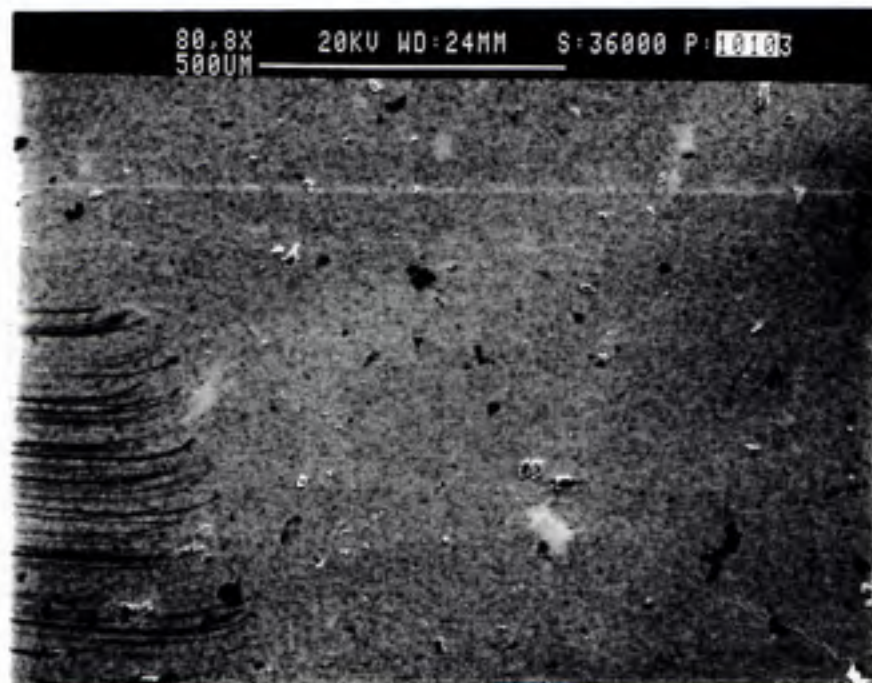


Figure 5-5b SEM Sample of Reclaimed Wastewater without Coagulation.

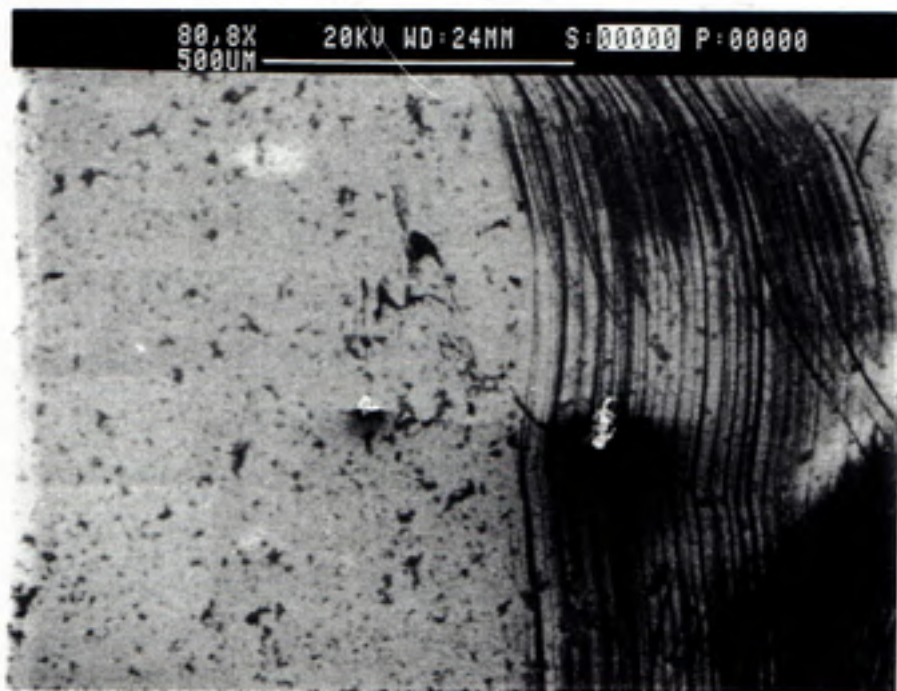
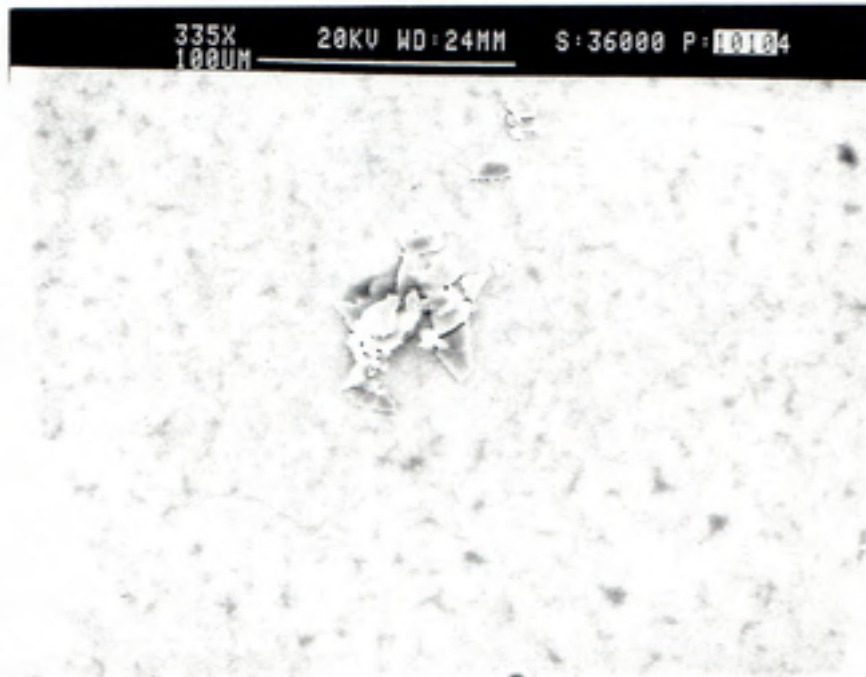


Figure 5-5c SEM Sample of Reclaimed Wastewater with Coagulation (12.5 mg/l Riverclear 3103).





2

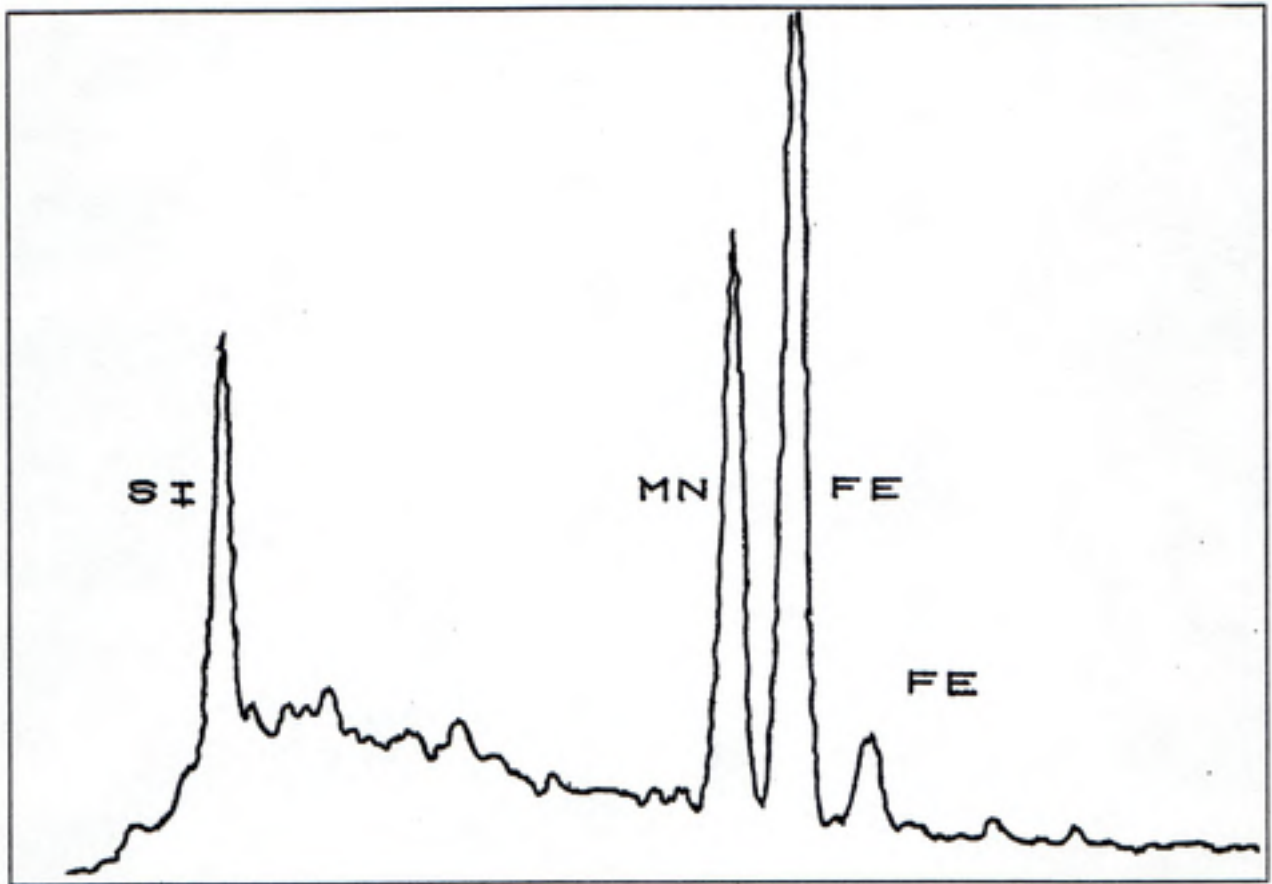
Figure 5-6 Magnification of Typical Particle Present in Reclaimed Wastewater.

QUANTEX X-RAY GRAPHICS

PR = S  
V = 512

28 SEC  
H = 20KEV 1:1Q

0 INT  
AQ = 20KEV 1Q



< 0.00KEV

XES

10.24KEV >

Figure 5-7 Energy Dispersive X-ray Analysis of Reclaimed Wastewater Particle

### Summary

The reclaimed wastewater is of very high quality based on conventional measures (COD, BOD<sub>5</sub>, and DOC); however, regrowth of microorganisms during the 15 h storage and in the National Spinning Company treatment units is expected. This problem could be addressed by application of additional chlorination and dechlorination stages in the National Spinning Company treatment process.

It is also evident that the reclaimed wastewater has the potential to stain the National Spinning Company fabrics. Applying a standard water treatment scheme of coagulation-flocculation-sedimentation to the treated wastewater after 15 h storage appears to significantly reduce the staining; however, some discoloration is still evident using the National Spinning Company staining test. The cause of this staining is not known. It is clear, however, that the reclaimed wastewater, without further treatment, will not be acceptable for use as process water in textile dyeing operations.

## CHAPTER SIX

### CONCLUSIONS

The following conclusions are made based on the results presented:

- The general indicators of wastewater treatment efficiency (BOD<sub>5</sub> and COD) suggest a wastewater of good quality was achieved. Storage of treated wastewater without additional of chlorine (or other disinfectant that will maintain a residual) caused regrowth of microorganisms.
- Results of SDWA Compliance Monitoring tests detected only trihalomethanes, (chloroform, bromodichloromethane, and chlorodibromomethane) and phthalates. THMs were less than 20 µg/l and well below the current MCL. Phthalates were well in excess of MCL (0.1482 compared to 0.004 mg/l); however, it is possible that phthalates are an artifact of the experiment.
- Reclaimed wastewater may cause staining of yarns produced at National Spinning Company even after coagulation with Riverclear 3103 polymer.

## CHAPTER SEVEN

## RECOMMENDATIONS

It is evident that the proposed treatment scheme at the City of Washington and the National Spinning Company will not produce the water quality required for dyeing operations. The primary concern with the reclaimed wastewater is the potential for staining of the textile products. Treatment technologies other than those proposed may result in removal of the problem contaminants. These include particle separation technologies such as micro filtration, which could be designed to remove bacteria as well as other particles of similar size. Further study into the nature of the reclaimed wastewater in terms of particle size distribution might indicate the potential for this treatment technology.

The use of reclaimed municipal wastewater as process water in textile dyeing operations has not been documented in the literature. Reclaimed water used for this purpose must be of very high quality to meet the process needs of the industry and to minimize the potential impacts on human health by inadvertent exposures. This report was intended

to investigate the quality of the reclaimed water from these perspectives.

It is important to note that the process water presently used by National Spinning Company is not regulated. However, use of reclaimed wastewater for this application would undoubtedly require that some minimum standards be met to protect the health of both National Spinning Company employees and customers. The decision to reuse wastewater, therefore, should be made cautiously with the full cooperation and knowledge of the responsible agencies and with an employee education program.

The experience and regulatory guidance on wastewater reuse is very limited in North Carolina and thus policy issues remain. Moreover, the lack of universal regulatory standards, limited experience, and the general public's less than favorable opinion towards wastewater reuse may concern National Spinning Company about potential legal issues. It is, therefore, imperative that the State of North Carolina work to develop a framework to regulate reuse projects as in other states (i.e., California). Undoubtedly, more innovative uses of reclaimed wastewater will be developed in the future in order to conserve water resources. Both the State and industry must look for reuse opportunities and develop the necessary technical framework to manage these projects effectively.

## REFERENCES

- APHA 1992. Standard Methods for the Examination of Water and Wastewater, 18<sup>th</sup> Edition. Washington D.C.
- Black & Veatch Design Memorandum, September, 1992. City of Washington, North Carolina Wastewater Facilities Improvements.
- Crook, J.; Tsang, K. R.; and Roberts, J. L. 1993. Water Reuse Criteria and Practice in the U.S., Proc. Dollars Down the Drain - The Potential for Water Conservation and Reuse in North Carolina; Triangle J Council of Governments, March 30, 1993, 1-52.
- EPA/625/R-92/004, September, 1992. Manual - Guidelines for Water Reuse.
- Goldstein, D.J.; Wei, I.; and Hicks, R.E. 1979. Reuse of Municipal Wastewater as Make-up to Circulating Cooling Systems, Proc. of the Water Reuse Symposium, Denver, CO, Vol. 1.
- Jeris, J. S. and Owens, R. W. 1975. Pilot-scale, High-rate Biological Denitrification, J. WPCF, 47:(8) 2043-2057.
- McCarty, P.L.; Beck, L.; and St. Amanti, P. 1969. Biological Denitrification of Wastewater by Addition of Organic Materials, Proc. 24<sup>th</sup> Ind. Waste Conf., Purdue Univ., 1271-1285.
- Miles, S. W. and Ridge, J. T. 1993. Water Conservation: It's Place in North Carolina's Water Resource Future, Proc. Dollars Down the Drain - The Potential for Water Conservation and Reuse in North Carolina; Triangle J Council of Governments, March 30, 1993, 55-80.
- Savage, E. S. 1985. Biological Denitrification in Deep-Bed Filters, Paper presented at the Georgia Water and Pollution Control Association, Savannah, GA, Aug. 12, 1985.

- Smith, S. A. and Guild, K. 1984. Development of Tuscon Metropolitan Wastewater Reuse Program, Proc. Water Reuse Symposium III - Future of Water Reuse, San Diego, CA, Aug. 26-31, 289-298.
- Sullins, C. 1993. Personal Communication, Workshop on Policies on Reuse of Municipal Effluents, Water Resources Research Institute of North Carolina, October 19.
- Sundberg, S. R.; Parker, J. D., Chapman, R, L.; and Rippon, D. W. 1991. Reclaimed Wastewater for Industrial Utilization, Symposium on Water Supply and Water Reuse - 1991 and Beyond, San Diego, CA, Jun. 2-6, 311-320.
- Upton, J.; Fergusson, A. and Savage, S. 1993. Denitrification of Wastewater: Operating Experiences in the US and Pilot-plant Studies in the UK, J. Inst of Wat. & Envr. Mngmt., 7:(1) 1-11.
- Water Pollution Control Federation. 1989. Water Reuse Manual of Practice, Second Edition. Alexandria, VA.
- York, D. W. . and Crook, J. 1991. Florida's Reuse Program Paves the Way, EPA 430/09-91-022, 34-38.