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RECOVERY OF CAUSTIC SODA FROM THE MERCERIZATION PROCESS

A THESIS

Presented to

The Faculty of the Graduate Division

by

Leonard Douglas Jones

In Partial Fulfillment

of the Requirements for the Degree

Master of Science in Textiles

Georgia Institute of Technology

July, 1965

RECOVERY OF CAUSTIC SODA FROM THE MERCERIZATION PROCESS

Approved:

Chairman

Date approved by Chairman:

*July 27, 1965*

Dedicated to

WILLIAM LEONARD HYDEN

*In life we shall find many men that are great, and some men  
that are good, but very few men that are both great and good.*

— C. C. Colton (1780-1832)

## ACKNOWLEDGMENTS

I would like to express my sincere appreciation to my thesis advisor, Dr. W. L. Hyden, to whom I owe a great deal for his valuable advice, guidance, and assistance. Appreciation is also expressed to Professor R. K. Flege and Dr. R. S. Ingols, who served as members of the thesis reading committee and offered many valuable suggestions and revisions.

Special appreciation is expressed to Dr. J. L. Taylor for his continued interest in my career. Without the financial assistance given, in the form of the T. E. Stribling Fellowship, this thesis and its succeeding publication would not have been possible.

The interest of the faculty and students of the A. French Textile School in this research has resulted in many valuable suggestions for which I am most grateful.

The research program conducted would not have been possible without the close cooperation of the firms assisting in the study. Foremost among the many prominent individuals to whom I am deeply indebted are Mr. John Newton and Mr. George Fogle.

In addition, the understanding and sufferance of my immediate family are warmly appreciated.

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## SUMMARY

The pollution of this nation's water resources has become a subject of great concern and study. Since it contributes significantly to industrial pollution problems, the textile industry shares in this concern and is progressing at a fast pace in the abatement of industrial pollution. Much effort has been expended during the past century toward the alleviation of textile effluent problems and the present trend seems to be one of a course of preventive measures. This study deals with the possible prevention of one of the problems faced by this industry.

The feasibility of recovering caustic soda from the process of mercerization of cotton fabrics was investigated in this study. Among the considerations included were the aspects of enforcement of process economy, and recovery as a means of reduction of water pollution. The economics of the recovery of caustic were investigated by the comparison of several methods of recovery. An attempt was made in the study to delineate the effect of the many process variables upon the selection and operation of a recovery system.

The actual mechanics of the installation of a caustic recovery system are discussed and the changes suggested in plant processes are those which would normally be made in the interest of economy and efficient operation of the processes. The result of the installation of a recovery system not only contributes to the reduction of the consumption of caustic soda but also contributes significantly to the reduction of plant water usage.

The proposals for the reuse of the caustic soda considered in this study employ the caustic in the recovered dilute form insomuch as possible to avoid costly reprocessing of the solution. Among the possible uses found for the dilute caustic solution were the processes of desizing, open boil-off, kier boiling, continuous scouring, bleaching and reuse in the mercerizing process.

The effect of the installation of a caustic recovery system upon the plant effluent quality and volume was investigated, with the effect found to be proportional to the production of the mercerizer and other plant processes. The specific significant effects were the reduction of the free alkalinity present and reduction of the volume of the plant effluent.

Included in the investigation were considerations of the treatment of textile wastes with chlorine and sodium hypochlorite, manufacture of sodium hypochlorite from the dilute caustic solutions and possible recovery of by-products from other plant processes.

In the recommendations of the study are specific proposals for further investigative work to determine the feasibility of processing textile fabrics within a partial, or closed, effluent recycle system. The difficulties which are foreseen are discussed with specific recommendations for avenues of attack of the major problems.

A comprehensive bibliography on the recovery of caustic from mercerization and the disposal of process effluents from cotton processing is appended as an addition to the references cited in the text.

## CHAPTER I

## INTRODUCTION

General

With the majority of the earth's surface covered by water and with man being constantly plagued by the destructive forces of flooding and erosion, it seems incongruous that society should be so concerned with the dumping of relatively small quantities of polluting substances. Yet, the pollution of the water resources is a problem, as attested by the recent statements of the President and pending actions of the Congress and various state legislatures. Then too, one must use caution, for the polluting is being done by the very industrial system which supports our present day high standard of living. But this concern is very real as is evidenced by the public declarations of politicians and the building into the basic foundation of many political platforms a continuing pledge to sustain and improve the quality of the nation's water resources. This country is not alone in its concern for what is probably its most vital natural resource. The problem is, by its very nature, within the scope of concern of all living beings.

The contribution of the textile industry to the contamination of these water resources has been the subject of much study throughout the past century. The emphasis which has been devoted to this subject of industrial water pollution may be attributed to the undesirable characteristics of the varied and peculiar type of waste discharged by the

textile industry. Much effort has been expended for pollution abatement and the present trend seems to be toward preventive measures. This research was concerned with the prevention, rather than the treatment of the liquid waste resulting from textile processing.

#### Mercerization

The process of mercerization in textile processing is an important and vital step which enables the natural fiber, cotton, to compete for markets which would otherwise be unattainable.

The effects of caustic soda on cotton were first observed in 1844 by an Englishman, John Mercer, who patented the process in 1850. However, it was not until after the effect of tension in the mercerizing process was discovered by H. A. Lowe in 1889 that the full import of this important discovery was evident. The term "mercerizing" is usually understood to refer to the treatment of cotton fabric with caustic soda (sodium hydroxide) in a sufficient concentration and under controlled tension so that a permanent high luster will be imparted. In addition to the development of luster on the fibers, other physical and chemical properties of the fiber are also altered. The term mercerization also implies, in a more general sense, the treatment of almost any kind of cellulose with almost any agent which will swell cellulose. The swelling of wood pulp by caustic, a step in the manufacture of viscose rayon is included. The process of mercerization did not become widely accepted until the latter years of the 19th century.

While there are many swelling agents which may be used on cellulose, caustic soda is the least expensive alkaline mercerizing agent

and is easier to control than most other swelling agents which have similar effects. The operating conditions of the mercerizing process do not have to be closely controlled as the action of the caustic cannot go as far as that of 62 per cent sulfuric or 70 per cent nitric acids which will seriously degrade the cellulose structure, or as that of Triton B (a tetra alkyl ammonium hydroxide) which can also dissolve cotton. Water and caustic will swell cellulose in dilute solutions but the effects of mercerization appear first at a concentration of approximately 14 per cent. Commercial mercerizing baths will usually range from 20 to 25 per cent concentration for treatment of dry fabrics and 40 to 55 per cent for wet fabrics to insure a sufficient and uniform penetration.

#### Statement of the Problem

The chemical, caustic soda, does not combine permanently with the cellulose but rather is washed out once the desired effect has been obtained. It is the removal of this chemical from the fabric and the subsequent disposal of the waste wash liquor which causes a water pollution problem. The pollution effect of the wash liquor for 1,000 pounds of fabric equals the effect created by a population equivalent of 60 to 80 people and will produce a waste water volume of 27,900 to 36,950 gallons per 1,000 pounds of goods (1). While the organic solids content of the wastes (600-1900 ppm) is not extremely high, the high alkalinity (approximately 2000 ppm of sodium bicarbonate) of the discharged solutions has a detrimental effect upon the waste treatment process (2). The caustic is used in such large quantities that the pH

of the effluent from the mercerizing process will average about 11 with a maximum of about 12.5. With a pH value of 10.5, the rate of biochemical oxygen demand is markedly reduced and greatly affects the decomposition rate of the waste (2, 3). Thus the main source of contamination arising in mercerizing is the free alkali which is associated with the processing of the cotton.

The final structure of the cellulose does not contain sodium or any product derived from it, and it is therefore evident that the entire amount used is either neutralized or is passed off into the wash waters. Since the caustic entering into combination with the cellulose does not undergo any permanent change, it is all theoretically subject to recovery. As caustic soda is the largest item in the cost of mercerizing, the idea of caustic recovery for repeated use is attractive from a standpoint of process economy.

From this general discussion two reasons for the recovery of caustic soda from the mercerization process develop. The first, and perhaps most appealing reason to management, is to economize and cut costs. The second is to avoid the pollution of the water resources. Another or third reason can be derived from the first two is that by the recovery of the caustic to cut costs and avoid pollution problems, the cost of waste treatment may also be reduced due to the elimination of a large amount of the free alkalinity in the plant effluent.

#### History of Caustic Recovery

With the advent of large-scale mercerization around the turn of the century, the costs associated with the process became an item of



close scrutiny. Caustic recovery probably had been practiced previously but the first published communications concerning the subject appeared in the years 1907-1908 (4). For a period of about 20 years extensive studies were conducted and several articles and patents were published concerning caustic recovery through various methods (6, 7, 8, 9, 10, 11). In 1937 Perry reviewed the subject of caustic recovery (12) and in 1941, Riere discussed the recovery of chemicals from industrial processing (13). However, it was not until after War II when many of the Western governments began to concern themselves again with the normal problems of society that the subject again emerged as a problem.

In 1946 Cherkinskii published an article dealing with the recovery by evaporation (14). A patent was issued to Steele and McMahon in 1953 (15) in favor of Allied Chemical Corporation, a company that has been one of the pioneers in the recovery of caustic from textile processing. Since that time there have been a number of general articles published, some of which include specific examples of caustic recovery systems (16, 17, 18, 19). Ray published an article in 1953 discussing recovery by evaporation (20) and Nemerow and Steele discussed the commercial applications of dialysis of caustic solutions in 1955 (21).

The process of mercerization became so important that Edelstein and Cady published a general bibliography of the literature concerning the process in 1937 (22) and Edelstein conducted a general study of the physical chemistry of mercerizing (23). Now in more recent years, the full impact of some of this earlier work is being felt. With better instrumentation as outlined by Ridley (24), much time and expense can be saved in this one process. Specific control features mentioned are

specific gravity, temperature, water levels and flow rates.

There have been many methods of caustic recovery proposed and actually put into use. The first step in any recovery system is the recovery of the effluent ensuing from the process, in this case, mercerization. The wash solution must be as concentrated as possible consistent with good efficient washing of the goods being processed. The counter-current flow technique so familiar in chemical engineering is normally used to recover the caustic solution under these conditions.

A plant doing relatively small amounts of mercerizing and a large volume of kier boiling will have the simplest recovery system. The dilute caustic solution from the mercerizer washing process can simply be transferred to a storage tank and then be used in this form in the kieriing process.

Schroeder (25) proposed another process for the reuse of the recovered caustic in its dilute form by the treatment with chlorine gas. The method never gained wide acceptance in spite of the simplicity and cheapness of the treatment. Theoretically one gram of chlorine will combine with 1.15 grams of caustic soda, but there will be some loss due to the action of the chlorine upon any organic impurities present in the recovered solution.

Other methods which have been suggested appear to have been more novel and ingenious than practical as none has ever gained wide acceptance. Goldthwait (26) reported on several of the methods in 1925. One suggestion was to neutralize the caustic solution with hydrochloric acid and electrolyze the resulting salt solution back to the caustic. The use of carbon dioxide to carbonate the caustic in the

fabric and reusing the carbonate as soda ash has been suggested. The advantages of this type of treatment would be the lack of need for the "acid sour," or neutralization of the residual caustic in the fabric, and the ability to obtain the carbon dioxide from the plant smokestack. The use of a steam boiler to continually evaporate the water into steam to be used in kiering was proposed in 1907. The concentrated caustic solution was to be recirculated through the mercerizer. Evidently the process was not practical, as no commercial applications were ever published.

#### Purpose of the Research

The recovery of caustic soda is a chemical processing operation carried out in an unusual place. In the process of this recovery operation there are many factors which exist that are not normally considered in the manufacture of caustic because of the very nature of the source of the raw material. The purpose of this research was to examine scientifically these unusual factors, devise an approach and design to accomplish the operation and delineate some of the many alternatives to be considered both from a scientific and economic outlook. The research paper was divided into several phases or areas of interest. In relating the process of mercerization to water pollution problems it was desirable to know the effects of the mercerizer upon a plant's effluent and any contributions made to the waste effluent by the process. Also the latter information was necessary prior to the design of any caustic recovery system.

## CHAPTER II

### THE RESEARCH

#### Description of the Research

The general scope of the problem studied necessitated the use of a somewhat broad approach while at the same time pursuing specific promising lines of attack. Therefore the research objectives were divided into four topics of interest which would lead to objective conclusions concerning the recovery of caustic soda. The four avenues of attack selected were: (a) a survey of the literature and current practice within several of the local plants; (b) a study of the caustic recovery process; (c) a study of a cotton finishing plant's effluent; and (d) an investigation of the process of recovery and possible reuse of the caustic solution.

#### Survey of Current Practice

Contact with industry, governmental agencies and other interested individuals was made to determine the current practices of waste treatment within the local textile industry (within the state of Georgia). The literature was consulted for the more general concepts and for specific data which would assist in clarification of the problem.

#### Recovery of Dilute Caustic

A study of the mercerization process to include recovery or disposal of the dilute caustic solution was made at a local plant.

This study included the generation of a proposed design to recover and store the dilute caustic wash solution. Engineering and economic factors were considered in the proposed process changes with an attempt made to form an objective conclusion which could be extended to cover the economics of caustic recovery for a typical plant.

#### Study of Plant Effluent

During the research period observations were made on the plant's effluent as to specific waste volumes and properties. Also included in this portion of the research was an attempt to isolate and identify substances in the effluent from the mercerization process which could interfere with further processing or possibly prove valuable as a by-product. The volume and properties of the waste were related specifically to the mercerization process with an estimation of the relative costs of treatment.

#### Recovery and Reuse of Caustic

An investigation was conducted into the economics of recovery and possible reuse of the caustic by various means with specific data to support the methods studied.

#### Instrumentation and Equipment

In addition to the fixtures, tools and glassware found in the normal chemical laboratory, there were a number of devices and instruments used which are listed below:

- a. Beckman Glass Electrode pH Meter, Model H 2.
- b. Soxhlet Extraction Device.

c. Perkin-Elmer Spectrophotometer, Model 221, with associated fixtures and tools.

d. 15-Ton capacity Hydraulic Press with Vacuum Pump.

Other devices described in the research were fabricated from items which are found in the normal chemical laboratory or a local hardware store.

#### Procedure

The laboratory analysis portion of the research was carried out using standard methods such as those published by the American Public Health Association (27) and the American Association of Textile Chemists and Colourists (28). Observations in the field were made at frequent intervals but not on a scheduled basis. The observations were usually in the afternoon of a normal workday.

#### Sampling Procedure

The sampling points selected and used in this research were: (a) a point approximately 100 yards from the discharge point of the plant; (b) the outfall pipe of the settling pond; (c) a point approximately 200 yards upstream from the settling pond; (d) a point approximately 0.5 miles below the outfall pipe of the settling pond; and (e) the wash liquor taken directly from the wash box discharge pipes of the mercerizing range within the plant. Upon arrival at the sampling points, the normal procedure was to note the relative flow of the effluent, the temperature of the solution, outside temperature, and physical appearance. The samples were collected and sealed in airtight containers for further analysis in the laboratory.

### Sample Preparation for Analysis

The samples taken from the mercerizing process were prepared for analysis by separating the organic portion, which was analyzed by the method of infrared absorption spectroscopy, from the inorganic portion, which was analyzed qualitatively by conventional chemical analytical techniques (29). The separation was accomplished by thoroughly mixing the sample with an organic solvent and withdrawing the organic portion through a separatory funnel. The organic portion of the sample was then distilled into several fractions over the boiling point range and analyzed as separate samples on the infrared spectrophotometer. A drying agent was used as the sodium chloride cells on the spectrophotometer are deteriorated by water. The inorganic portion of the sample was analyzed for the expected substances by semi-micro analytical techniques.

### Chlorination Procedure

The preparation of the samples involving the use of chlorine were prepared by absorption of chlorine. Chlorine gas was bubbled through a column containing the sample. To assist the reactions and the solubility of the chlorine, the tube was immersed in water bath which was maintained at a temperature of 65-75 degrees Fahrenheit. After the completion of the reaction, the sample was allowed to stand at room temperature for a minimum of 12 hours before use.

## CHAPTER III

## DISCUSSION OF RESULTS

Survey Data

The disposal of the liquid wastes created as a result of textile finishing operations generate problems of great and growing concern. Much study is needed for solutions to these problems. The alkaline waste wash solution from the mercerization process represents only a small (but significant) portion of the total waste problem. This fact was recognized early in the research and the survey of the local industry conducted by the author only served further to emphasize the point. These investigations did bring several factors to light which should be commented upon since they could be of considerable aid to other investigators and will serve to further clarify certain points brought out in later portions of this research.

The results of the local survey indicated that while many of the firms in the state of Georgia are very conscious of their moral and legal obligations in the disposal of their waste products and are forging ahead in this respect, others may not be considering fully these requirements and the full implication of the detrimental consequences of the polluting effects. In one case, a firm has investigated a unique process of partial treatment by chlorine gas, which seems to have considerable merit. Even though the results of this work have not been published, the author investigated the process further. The effective-



ness of this process, the use of chlorine in the treatment of colored wastes, is discussed later in connection with the experimental phases of this research. Other plants give the highly polluted effluent only a token treatment of settling before discarding directly into the stream or sewer. In the most aggressive and highly productive plants, one finds the situation first mentioned but also in addition, the firms are actively attempting to salvage process chemicals through process by-product and chemical recovery. Not only do these actions significantly decrease the polluting effect of the process and plant effluent, but also give the firm a production cost advantage which was previously impossible to attain.

Through the recovery of chemicals, in this specific case, caustic soda, the effluent volume and quality is changed through reduction of wash water volume and removal of a considerable portion of the free alkalinity which was previously wasted. The desirability of accomplishing this reduction in the plant studied was initially pointed out by Ingols and Sharp (30) in an independent investigation.

The pilot plant study indicates that large BOD reductions are possible with a plastic film filter and secondary lagoon. The drop in effectiveness of the system as the recirculation ratio decreases emphasizes the importance of a lagoon which can act as a reservoir of lower pH water. It should be emphasized that reducing the pH or total alkalinity of the raw waste by implant modifications should increase the overall effectiveness of the filter-lagoon system according to the pH data. The filters are capable of much higher BOD loadings with effective removals in studies conducted at normal pH values with other wastes.

In a previous study by Morgan (31), it was found that the polluting load of the waste was in the order of 900 to 1150 parts per million BOD. With the large amount of free alkalinity present, an average reduction of only

52 per cent of the total BOD was realized at an economical cost. In analyzing the data presented by Morgan, it was found that, in the waste treatment process under investigation, the cost of the acid for neutralization constituted 67 per cent of the total cost of treatment. Therefore, any reduction in the causes of this high alkalinity will significantly reduce the waste treatment cost. In further studies of this highly variable waste, Pierce (32) continued the work of Morgan in further development of the magnesia process for treatment of alkaline textile wastes. The results of the work conducted by Pierce indicated that it is feasible to treat the wastes of the specific plant at a cost of \$30 to \$50 per million gallons with a final sludge volume of only 0.5 per cent of the total volume of waste treated. The clarification obtained at this cost was: 27 per cent reduction in Biochemical Oxygen Demand (BOD); 95 per cent turbidity removal; and 50 per cent color removal. Again it must be noted that the cost of the acid for neutralization of the effluent constituted over 50 per cent of the total cost of waste treatment. Pierce recommended that some other means of pH reduction be investigated as an alternative to the high cost. These factors, plus the fact of the cost of the caustic soda (\$3.05 per 100 pounds) discarded in the process, makes process chemical recovery most attractive.

### Mercerization as a Process

#### Plant Observations

The major portion of the work in the study of the mercerization process consisted of a combination of field observations, analytical

laboratory tests and designs for modification of equipment and processes. The mercerization equipment used in the plant consisted of a three-roll caustic pad box, a variable width tenter frame and seven wash boxes which were connected for parallel flow of the washing solution. The fabric was mercerized in the dry form which entailed saturating the cloth in an approximately 18 per cent caustic solution, squeezing to remove excess pad liquor, pulling out to width, holding under tension for the length of the tenter frame and washing the fabric in the seven wash boxes. The process was controlled through concentration of caustic in the pad liquor, cloth absorption at the pad, tension both as to width and length throughout the tenter frame and wash boxes, rate of flow and temperature of wash solutions, and overall production rate of the entire assembly.

The production rate of mercerization equipment is, as a general rule, dependent upon the weight and character of the fabric processed. The production figures are not given in this research as this represents information in which the plant management has a proprietary interest and would not wish to make public. In brief, the fabric was boiled off, and possibly bleached, and processed at a rate commensurate with economical production and efficient washing capacity.

In an analysis of data developed by the plant in an independent study (33) of the washing system, several items were uncovered which should be discussed. Samples of fabric were taken by the plant personnel at each stage of the process and analyzed for caustic content prior to and immediately after each step. The data thus generated were plotted by the author, resulting in the graph as shown in Figure 1. From this

Cloth Content.  
Average Per Cent  
NaOH Leaving Each  
Wash.

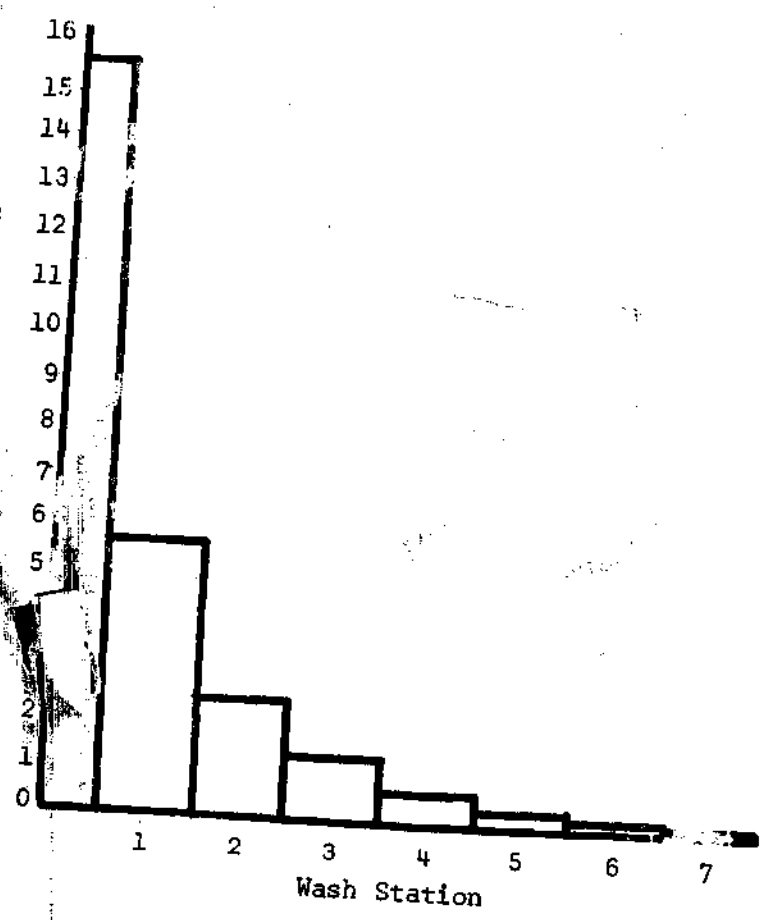


Figure 1 Cloth Caustic Content Leaving Each Wash

curve it can be deduced that the washing efficiency (relative percentage of caustic removed) of the washing station is approximately the same and will range from about 55 to 65 per cent. In further analyzing this data, it is found that when the marginal contributions of each washing station are plotted as in Figure 2, there was a very small marginal benefit gained between the fourth and fifth washing station and there was little, if any, gain beyond the fifth washing station. Perhaps a more meaningful and effective representation is cumulative per cent removed from the fabric as is shown plotted in Figure 3. These factors were considered in the design and presentation of a new washing system design (See Appendix).

#### Field Observations

At various times in this research, it was desirable to know the effect of the process studied in relation to the total waste discharged by the plant. Therefore, observations of the plant effluent were made during each visit to the plant and an attempt made to correlate these findings with the processes producing the wastes at the specific time. It was found during these visits that the mercerizer did contribute significantly to the volume and quality of the plant effluent. These findings were further substantiated by Brown's observations (34) that caustic waste constitutes about 19 per cent of the total volume, 37 per cent of the BOD, and 60 per cent of the total alkalinity. In this research, the mercerizer was found to contribute approximately 30 per cent of the total waste volume while operating. However since the mercerizer normally operates for less than two shifts per day, it was found that the actual total daily contribution was more on the order

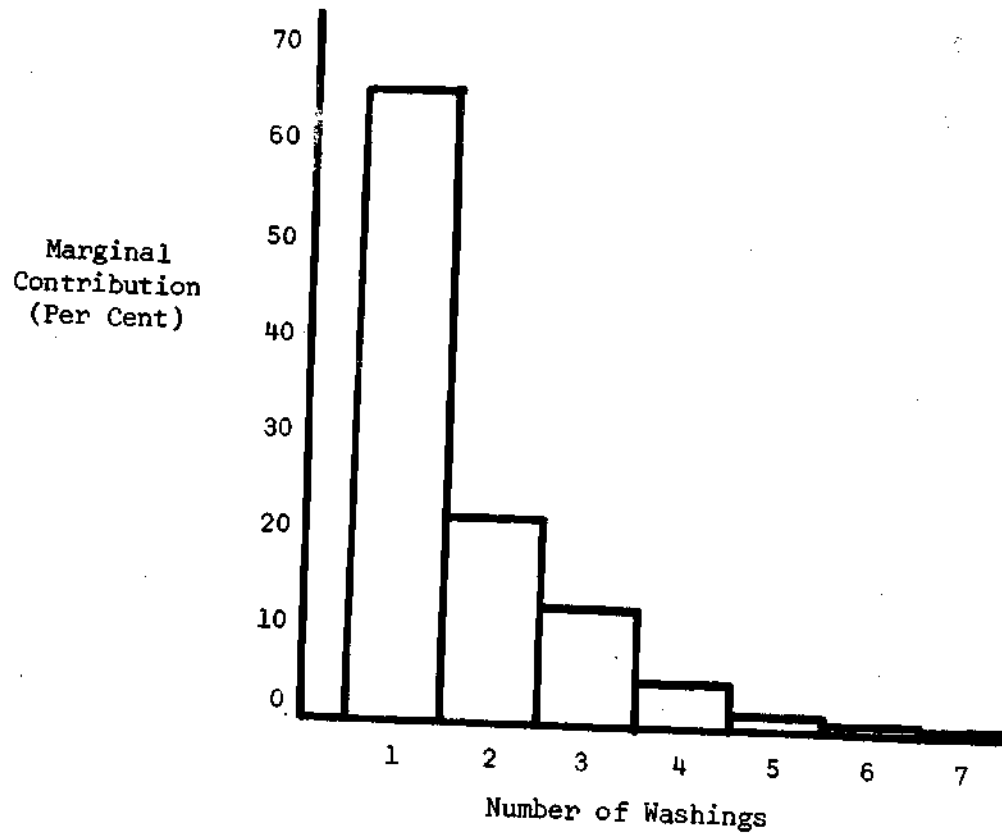


Figure 2. Marginal Contribution of Washing Stations

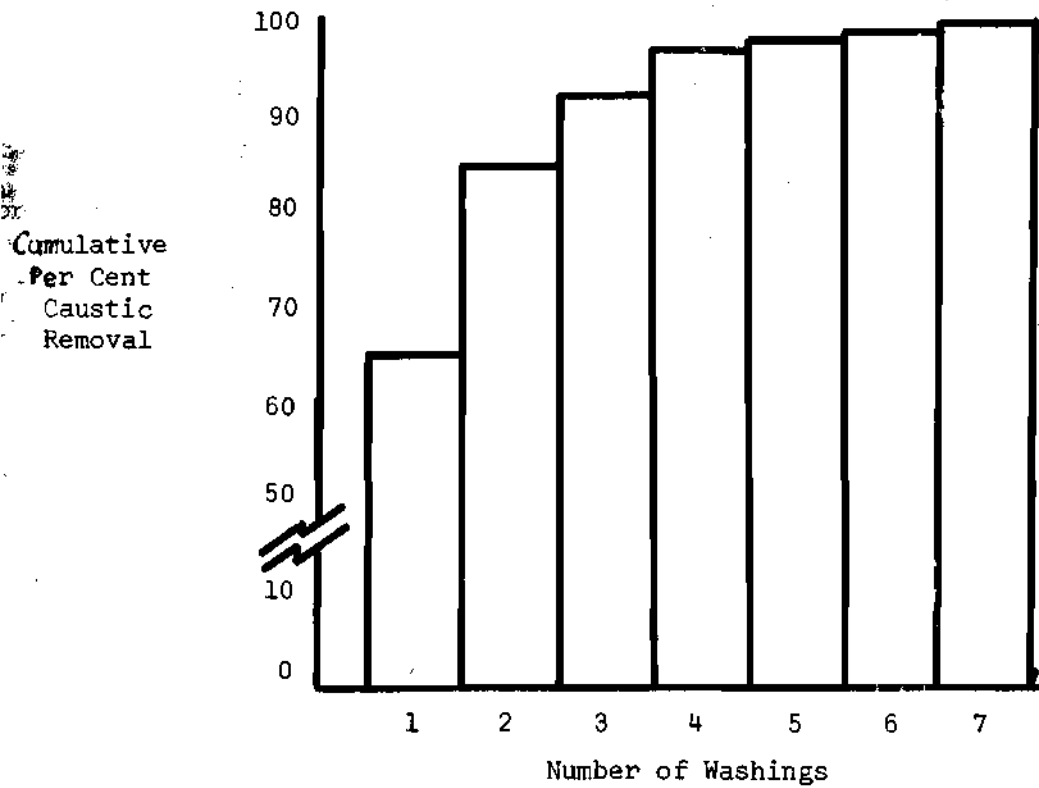


Figure 3. Effect of Number of Washings on  
Cumulative Per Cent Caustic Removed

of 17 per cent of the total volume. The remaining 2 per cent difference between the observations of the author and the findings of Brown could conceivably come from other sources within the plant. Even if this were not so, the difference was not considered significant as the physical make-up of each textile finishing plant is highly varied.

#### Mercerizer Wash Solution Analysis

The analysis of the organic extract from the mercerizer wash solution revealed that the majority of the impurities present were short chain aromatic and aliphatic nitrogenous alcoholic and sulphated compounds. The infrared spectra contained relatively strong but not sharply defined peaks at wavelengths of 8.75 and 8.99 microns. The strongest absorption range noted was in the area of 8.20 to 9.25 microns and 12 to 14 microns. Other areas of absorption were in the area of 6 to 7, 13 to 14, and 15 microns. These absorption areas are characteristic of the -C-C-C- and aromatic bonds, and the  $\text{CNH}_2$ , C-OH, and S=O polar groups. Characteristic spectra are reproduced in Figures 4, 5, and 6. The solvents used in the analysis were carbon tetrachloride and carbon disulfide. In the actual plotting of the spectra, a blank cell containing the pure solvent was put into the reference beam of the spectrophotometer to cancel the absorption characteristics of the solvent. All spectra plotted were calibrated using one of the sharp absorption peaks from the spectra of polystyrene as a reference point.

The inorganic portion of the solution was analyzed using semi-micro chemical analytical techniques (29). The cations and anions found present were: (a) strong indications for calcium, sodium, carbonate, and sulfate; and (b) weak indications for iron, ammonia, and



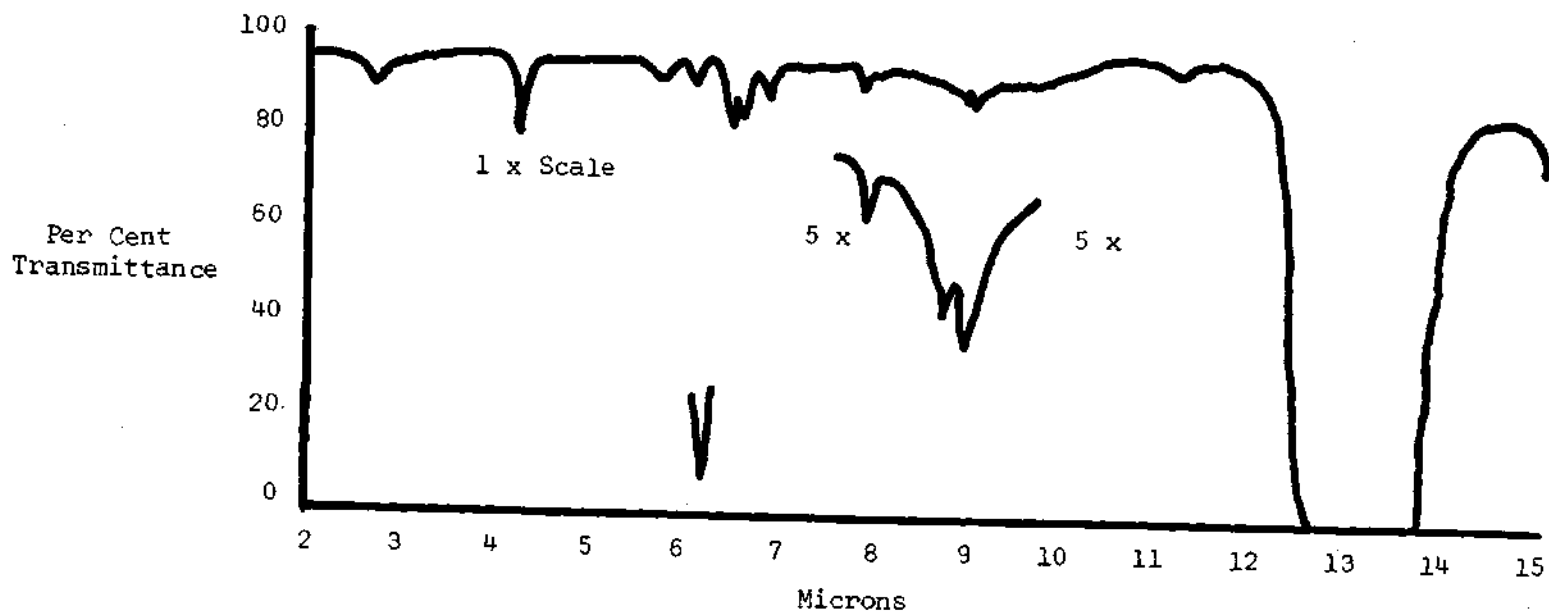


Figure 4. Characteristic Infrared Spectrum of Mercerizer Wash Solution Extract

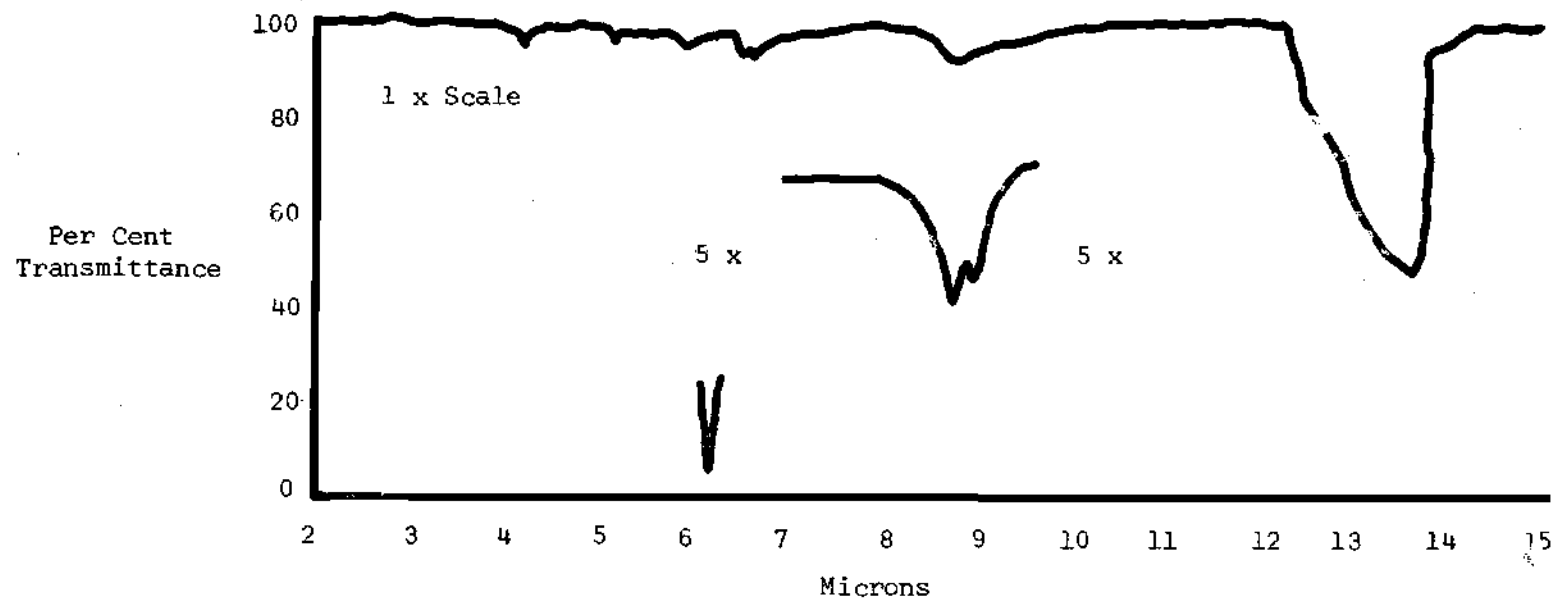


Figure 5. Characteristic Infrared Spectrum of Mercerizer Wash Solution Extract

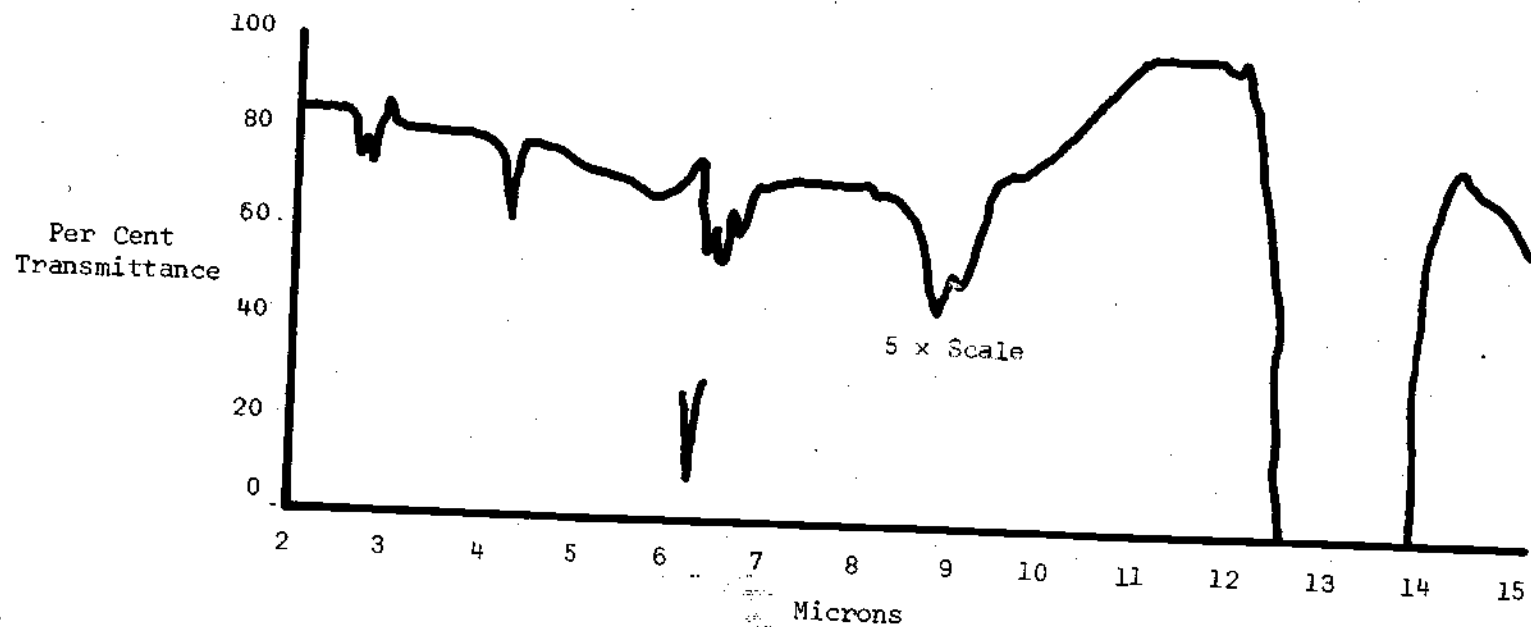


Figure 6. Characteristic Infrared Spectrum of Mercerizer Wash Solution Extract

nitrate. There were also traces of the divalent sulphide ion. All of the ions found as impurities in the solution were expected to be present but the analysis gave a positive verification of their presence.

#### Economics of Recovery

The consideration of recovery of caustic soda from the mercerization process involved several separate steps which were considered independent of one another. These steps were:

- a. Improvement of washing efficiency.
- b. Recovery and storage of dilute wash solutions.
- c. Reconcentration and purification of recovered caustic.
- d. Possible reuse of both the dilute and reconcentrated solutions.

The existing situation in the plant under study was to discard the spent washing solutions directly into the sewer with no attempt made to reclaim any of the solution. Thus the range of possibilities extend through the use of a total recovery concept. In the design of a recovery system, it was considered desirable to maintain, if not improve, the efficiency of the existing washing system. Therefore, the following features were incorporated into the design presented to the plant management (See Figures 11, 12, 13, and 14).

- a. The use of steam directly on the surface of the fabric to assist in penetration of the wash water and removal of the tenacious caustic solution.
- b. Employment of the counter-current cross-flow technique to expose the fabric to progressively lower concentrations of the caustic

solution and economize on the amount of water used.

c. Control of the rate of flow of the wash solution from the first wash box in the line (discharge point for the wash solution) to allow some control over the caustic concentration of the recovered solution.

d. Use of removable baffles in the wash boxes to establish the cross flow. This feature also facilitates threading up of the fabric when the baffles are removed.

e. Routing of the recovered solution through a heat exchanger to cut down on the amount of heat lost as a result of the process.

In the design of the washing system, there were numerous features which could have been incorporated into the system. It was decided to present the selected design because of the desirable features already noted plus the low initial capital investment required by the use of a gravity flow system.

The economics of the recovery of caustic soda by evaporation were investigated and the results plotted as shown in Figure 7. These results were calculated from the operating data of this specific plant and would be slightly different in another plant. The base prices used were obtained from commercial manufacturer's data and could not be taken as typical.

The delivered cost of caustic soda was taken to be \$71 per ton which is the commercial rate plus the freight and unloading costs involved. The depreciation period was taken as ten years with the cost of floor space, electricity and steam estimated on the best available information.

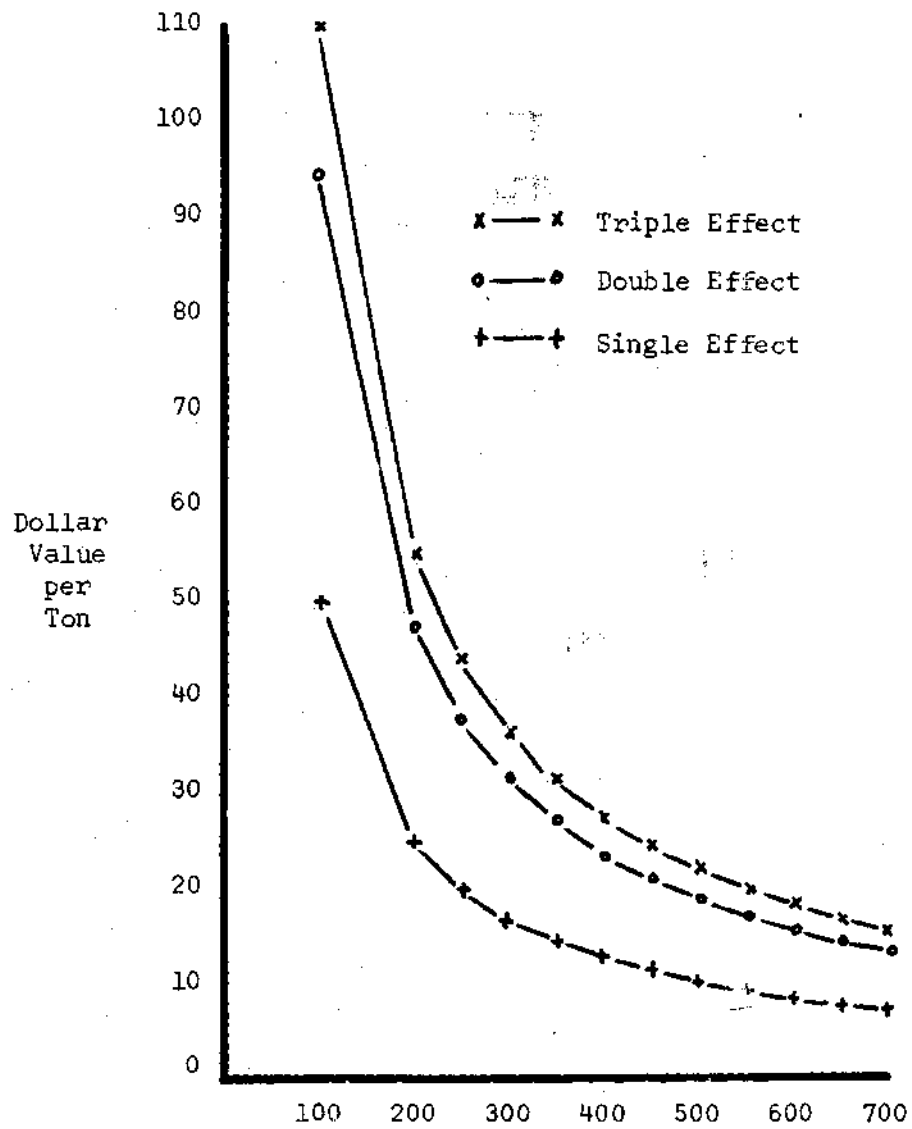


Figure 7. Relation of Cost per Ton for Various Evaporation Methods

At the present production level, the plant may recover approximately 400 tons of caustic soda annually for a reduced cash outlay of \$28,400. However, this savings cannot all be realized as the cost of recovery and reconcentration are in the order of magnitude of \$12.50, \$23.80 or \$27.50 per ton depending upon the type of evaporator (single or multiple stage) used and the final concentration and purity of the recovered solution. It was evident from the data that if a means for use of the dilute caustic without reconcentration was found, the attractiveness of the proposal for recovery would be further enhanced.

Possible points of reuse of dilute caustic solution were found in the following processes:

- a. Dilution of concentrated (50%) caustic to supply the caustic saturator on the mercerizer.
- b. Open boil-off progressive jig.
- c. Scouring.
- d. Bleaching.
- e. Manufacture chlorine bleach (not presently practiced at the plant).

The installation of a more efficient washing system (such as that proposed) will result in an effluent volume from the process of approximately 8,000-14,000 gallons per day at an average flow rate of 14 gallons per minute during the operating period. With this washing system, the concentration of the recovered solution will be approximately 5 per cent. The solutions from the existing system averaged approximately 3.5 per cent even with the few close controls present on the washing system. It is the author's opinion that one may expect

the recovered solution to contain at least 5 per cent caustic. However, even if this opinion were in error, the total amount of anhydrous caustic reclaimed is constant with only the volume of the solution variable. At a price of \$71 per ton delivered cost, this solution which is discarded contains an average of 3,200 pounds of caustic per day and represents a value loss of approximately \$113.50 per day. The use of any portion of the solution in its dilute form represents a true saving of at least the marginal value of the caustic involved.

Dilute caustic liquor (5 per cent) could be used at the approximate rate of 2,500 gallons per day to dilute the fresh 50 per cent caustic to a concentration of 20 per cent for use at the saturator pad of the mercerizer. There is some chance at this point of the impurities present in the recovered solution interfering with subsequent processing. However, the experimental work which was conducted in some earlier studies by Edelstein (35) indicate that most of these fears are probably unfounded. It is desirable to have the caustic pad solution as pure as possible but small amounts of impurities do not seem to adversely affect further processing. The use of the dilute caustic solution for this purpose would represent a saving of approximately 1,100 pounds of anhydrous caustic soda at a cost value of \$39 per day. On an annual basis, the savings represented by this use alone would amount to approximately \$9,750.

The processes of scouring and open boil-off progressive jig utilize caustic soda in a range of concentrations of 3 to 6 per cent, dependent upon the fabric processed. The caustic liquor may be used also in the washer in front of the caustic saturator of the J box in



the scouring process. Since the production figures for the scouring process were not available to the author, a reasonable estimate of the operational data was made based on observations made while in the plant. It was estimated that the scouring process would operate one shift per day for a total of 125 working days per year. The progressive jig was estimated to operate 75 working days per year. Based on these conservative estimates, a feed rate of 5 gallons per minute and the dumping of one 500 gallon tank per day from the open boil-off, the dilute caustic liquor used will be, on an annual basis, approximately 518,000 gallons representing 114 tons of anhydrous caustic soda and a dollar value of \$8,080.

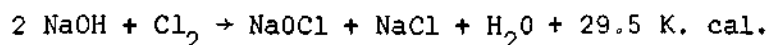
Other processes in which caustic soda can be used at concentrations of 7 per cent or less are desizing, adjusting pH of peroxide bleach bath and dyeing. Data on which to base reasonable conclusions were not readily available for these processes and there were thus no attempts made to estimate the quantities involved in these processes. However, the processes normally do not consume a large portion of the caustic requirements and any attempt to use caustic which is contaminated in the dyeing process would meet considerable resistance from experienced plant personnel because of the inherent problems involved and the high penalty cost of a poor dyeing. From discussions with plant personnel, it was deduced that it would be feasible to use reclaimed caustic in dyeing only if it were filtered to remove the bulk of the impurities.

The calculations made using figures generated by plant observations assume that the production rates used will remain constant. Realizing that in a practical situation, the assumption will not hold,

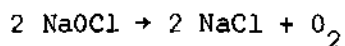
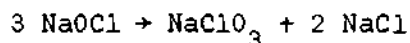
it must be noted that the flow routes of the dilute caustic should be reviewed periodically with alternate economic considerations in mind. The total quantity of the suggested reuse of the dilute caustic liquor involved will give an average daily consumption of some 5,000 gallons of the 5 per cent liquor representing an average daily saving of \$78 per day or on an annual basis, \$18,830. These cost figures represent only the direct process savings resulting from the reuse of the spent caustic solution.

The economics of the considerations for reconcentration by means of evaporation change as a result of the reuse of the dilute liquor. In referring to Figure 7, one finds that the fixed cost of evaporation per ton of caustic reclaimed increases sharply as a result of the withdrawal of the 265 tons from the recovery system. At this point, reconcentration to the desired level by evaporation by any means other than with a single effect evaporator becomes uneconomical. The calculated break-even point for the three systems of evaporation was 71, 134, and 155 tons of caustic with a delivered cost of \$71 per ton.

These comparisons made investigation into other possible uses of the remaining effluent sufficiently attractive from an economic point of view to warrant consideration of the manufacture of chlorine bleach. Chlorine bleach, sodium hypochlorite, is manufactured commercially by the absorption of chlorine into a caustic solution according to the following reaction:



The reaction is exothermic (generates heat) which has an adverse effect upon the bleach solution obtained by the generation of a further reaction of the hypochlorite to the chlorate and chloride. Thus:



Further, if an excess of chlorine is added, free hypochlorous acid will be formed resulting in an unstable bleach solution. In the manufacture of the bleach, the solution is cooled to a predetermined temperature by means of a heat exchanger and maintained below the temperature of 80° Fahrenheit with an excess of alkali kept in the solution (above a neutral pH). Theoretically, to absorb chlorine gas in a solution of caustic soda to full saturation would require 40 units of caustic soda for 35.5 units of chlorine. Commercially, the amounts used are approximately 125 pounds of caustic soda for 100 pounds of chlorine thereby allowing for a slight excess of alkali in solution. Since chlorine is available in the local area for \$3.25 per 100 pounds, the raw material cost for the manufacture of one gallon of hypochlorite bleach with an available chlorine content of 5.25 per cent (commercial strength) is \$0.0065 per gallon. If one considers that the dilute caustic is a waste product, the raw material cost then becomes \$0.0027 per gallon or the cost of the chlorine alone. In the manufacture of the bleach on a laboratory scale, some difficulty was encountered in gaining absorption of the chlorine into the dilute caustic solutions. The method

which seemed to work best was to introduce the chlorine into the bottom of a glass tube filled with the caustic solution. The commercial application of this method utilizes a tower absorption column approximately 15 feet in height and four feet in diameter equipped with a circulating type cooling coil and a gas diffusion device at the bottom of the tower (36). The strength of hypochlorite bleaching solutions used in a textile bleaching process usually contain 2.7 to 7.0 grams of available chlorine per liter corresponding to a commercial strength of .27 to .70 per cent available chlorine. Thus the bleach manufactured from the dilute caustic would be suitable for use if diluted to the proper concentration. Personal investigations by the author have shown that hypochlorite bleach retails commercially on the local market in one gallon lots within a price range of \$0.75 to \$0.85 per gallon and in bulk quantities at \$4.00 per 100 pounds with a potential dollar saving to a plant of \$0.0011 per pound of cotton goods bleached.

#### Relation with Waste Treatment

Morgan (37), Pierce (38), and Ingols and Sharp (39), demonstrated in their respective papers the effect of the high alkalinity upon attempts to successfully treat liquid wastes from this specific plant. Any decrease in the amount of alkalinity significantly affects the cost and effectiveness of the waste treatment. Correlating the field observations summarized in Tables 2 and 3 with the data obtained by Morgan (40) and Pierce (41), the effective unit cost of treatment is reduced to approximately \$36.45 per million gallons. This cost figure was based on the assumption that the cost of the equivalent amount of

sulfuric acid required to neutralize the caustic solution being recovered. was saved. The cost of sulfuric acid was taken to be \$22.25 per ton.

Table 2. Summary of Temperature Observations  
(Degrees Fahrenheit)

Sample Point	DATE 1965.						
	1/18	1/25	4/9	4/28	5/5	5/13	6/2
Tap Water	49		56				
Outside Air	56	60	82	78	83	88	90
Sample Point 1	108	110	110	110	105	100	118
1		108	110		105	110	117
2	94	98	102	100	100	102	108
3	68	89	96	92	92	99	99
4	54		68	67	66	65	

Not only will the cost of the waste treatment per unit volume be lowered, but since the mercerizer normally contributes to the flow rate of approximately 112 gallons per minute or a total of some 60,000 gallons of liquid caustic effluent per day of operation, the total waste effluent to be treated will be reduced by this amount. The total effluent of the plant presently discharges approximately 170,000 gallons per day and is thus effectively reduced by one-third with a consequent withdrawal of the associated caustic. If the waste effluent were treated by the magnesia

process, the reduction in volume has a potential annual dollar value of \$600. The annual savings in sulfuric acid costs would be in excess of \$20,000.

Nemerow (3) stressed the importance of segregation and separate treatment of characteristic wastes to achieve effective and economical waste treatment by either biological or chemical means. Prior to the consideration of caustic recovery, the use of the technique of waste separation was not deemed feasible (42). However, the field observations in Table 3 indicate that if the dominating effect of the mercerizer is effectively removed, the separation of the various types of waste is desirable if only from the standpoint of the possibility of neutralization by mixing.

The use of chlorine in the treatment of highly colored wastes was investigated with the results being summarized in Table 4. It is known from the basic chemistry of dyes and the literature (43) that the use of chlorine would prove effective in most cases. There are dyes, such as the vat classification, which are resistant to decoloration by chlorine. However, the chromophoric groups of those dyes are usually affected by alkali of which there is an abundant supply in the effluent. All of the colored wastes which were studied in connection with this research were cleared by treatment with chlorine. Since a high chlorine content adversely affects the biodegradation of the waste, the chlorination or final adjustment of the color would be accomplished in the latter stages of the waste treatment process. There were no quantitative tests undertaken in this area since the destruction of the color did not have a direct bearing on the recovery of caustic soda.

Table 3. Summary of pH Observations

Sample Point	D A T E						
	1/18	1/25	4/9	4/28	5/5	5/13	6/2
1			11.4	11.6	11.6	10.4	11.7
1			4.3		11.8	10.9	11.5
2			11.2	11.6	11.9	11.7	11.6
3			11.4	11.8	11.9	11.5	11.5

The bleach solution prepared from the dilute caustic solution was used instead of the chlorine gas to treat the highly colored waste with varying degrees of success. The results of the tests are tabulated in Table 5. The degree of success, in all cases, seemed to depend primarily upon the pH and temperature of the waste solution. These preliminary indications were substantiated by Hartsuch (44) in a general discussion of the properties and actions of bleaching solutions. Hartsuch pointed out that there is a rapid increase in the rate of bleaching action as the alkalinity is decreased from a pH of 13 to pH 11 with the rate remaining constant until a pH of 9 after which the rate again increases to a maximum at a pH of 7. The inverse effect happens from the acid side of the pH scale with the rate of reaction at pH 2 about the same as at pH of 8. From the acid side the rate of reaction decreases to a minimum at a pH of 4 and from that point increases again to a maximum at the neutral point. The fastest and most effective rates of reaction

are thus found in the pH range of 5 to 11.

Table 4. Treatment of Waste Solutions with Chlorine Gas

Sample Point	Date 1965	Temperature (°C)	pH	Remarks	pH After Treatment
1	4/9	28	11.4	Cleared	
1	4/9	28	4.3	Partially Cleared	
2	4/9	28	11.2	Cleared, Remained Turbid	
1	5/5	30	11.6	Cleared	6.7
1	5/5	30	11.8	Cleared	4.1
2	5/5	30	11.9	Cleared, Remained Turbid	6.0
3	5/5	30	11.9	Cleared, Remained Turbid	6.2
1	5/13	30	10.4	Cleared	6.5
1	5/13	30	10.9	Cleared	6.2
2	5/13	30	11.7	Cleared, Remained Turbid	5.8
3	5/13	30	11.5	Cleared, Remained Turbid	6.4

These facts developed by Hartsuch (45) were further substantiated by neutralizing a sample of the waste effluent at which point the minimum amount of bleach liquor was required to destroy the color. Thus the effectiveness of the chlorination or treatment of the waste with excess bleach will be enhanced by accomplishing this phase as the final step of the purification process when the solution is nearer the neutral



point. However, the volume of bleach liquor and temperature required to affect the color of the waste effluent is too high to consider as an economical means of decoloration (See Table 5).

Table 5. Treatment of Waste Solutions with Hypochlorite Bleach

Sample Volume (ml.)	Treatment	Remarks
50	Titrate to color change (30 degrees C.)	10.0 ml. changed the color from dark blue to pale yellow.
50	Neutralize with HCl. Titrate to color change. (30 degrees C.)	8.0 ml. changed the color from dark blue to pale yellow.
50	Heat to 45 degrees C. Titrate to color change.	9.2 ml. changed the color from dark blue to pale yellow.
50	Heat to 45 degrees C. Neutralize with HCl. Titrate to color change.	7.4 ml. changed the color from dark blue to pale yellow.

NOTE: Hypochlorite bleach characteristics:

Specific Gravity	1.080
pH	11.00
Per Cent Available Chlorine (KI)	8.6%

## CHAPTER IV

## CONCLUSIONS

The recovery of caustic soda from the mercerization process should be practiced in all textile plants conducting the process. This conclusion is drawn from the experimental and economic evidence presented in this research and applies to all plants.

The actual mechanics of the installation of the process of a caustic recovery system are changes which normally should be made in the interest of economy and efficient operation of the process. The results of the installation of a system such as that proposed in this research will not only contribute to the saving of caustic soda but also contribute significantly to the economizing of the plant water usage.

The dilute caustic liquor recovered should be used in the recovered dilute form in so much as possible to avoid costly reprocessing of the solution. The solution should be used primarily for the dilution of concentrated solutions at the mercerizer saturator pad, desizing, open boil-off progressive jig and kier boiling, if the latter is an integral plant process. Should excess effluent remain after the above uses, the excess could then be used in pH adjustment of the bleach baths and at the caustic saturator in the scouring operation.

After the uses for the dilute caustic solution have been exhausted, the effluent then still should not be discarded. If the re-

maintaining quantity available is sufficient to exceed the respective break-even points for single and multiple effect evaporation, then the appropriate evaporation system should be installed keeping in mind the future production plans for the plant. In addition to, or perhaps prior to, the installation of an evaporation system, the typical plant should investigate the possibility of the manufacture of hypochlorite bleach. The only disadvantage to this conclusion is the possible lack of adequate process control due to the unusual location of the chemical manufacturing operation.

The effect of the installation of a caustic recovery system upon the plant effluent quality and volume would be immediate with the direct impact being proportional to the production of the mercerizer and other plant processes. The specific effects would be the reduction of free alkalinity and volume of plant effluent.

The investigative area of work of a preventive nature with respect to water pollution in the cotton textile industry is a very fertile area in which considerable progress must be accomplished within the next decade. Much preliminary work has already been completed but there are many basic investigations which should be immediately explored in depth.

## CHAPTER V

## APPLICATIONS

The normal textile plant engaged in the finishing of cotton fabrics, or almost any type of fabric, normally uses water in a "one-shot" system. The water is used in one process and immediately discharged into the sewer or stream. This practice is represented by the flow chart shown as Figure 8.

An ideal system would be one in which there is no effluent from a finishing plant and fresh water is used only to replace losses due to the normal attrition caused by fabric take-up and evaporative (drying) losses. The quantity of water required to satisfy peak load demands is an integral requirement of the system and is held either in the treatment area or in temporary storage until needed. Such a system is envisioned operating in conjunction with process by-product recovery and is not possible without the by-products being withdrawn at predetermined points. The investigative work completed in this research has led the author to conclude that the proposal of a type system is extremely attractive and may in time become mandatory. The process flow for the ideal system is represented by the flow chart outlined in Figure 9. The impact of the successful implementation is the marked reduction and possible elimination of the liquid polluting effects of the largest industry in the state of Georgia. In addition, the venture will be financially rewarding to each manufacturer thus enabling

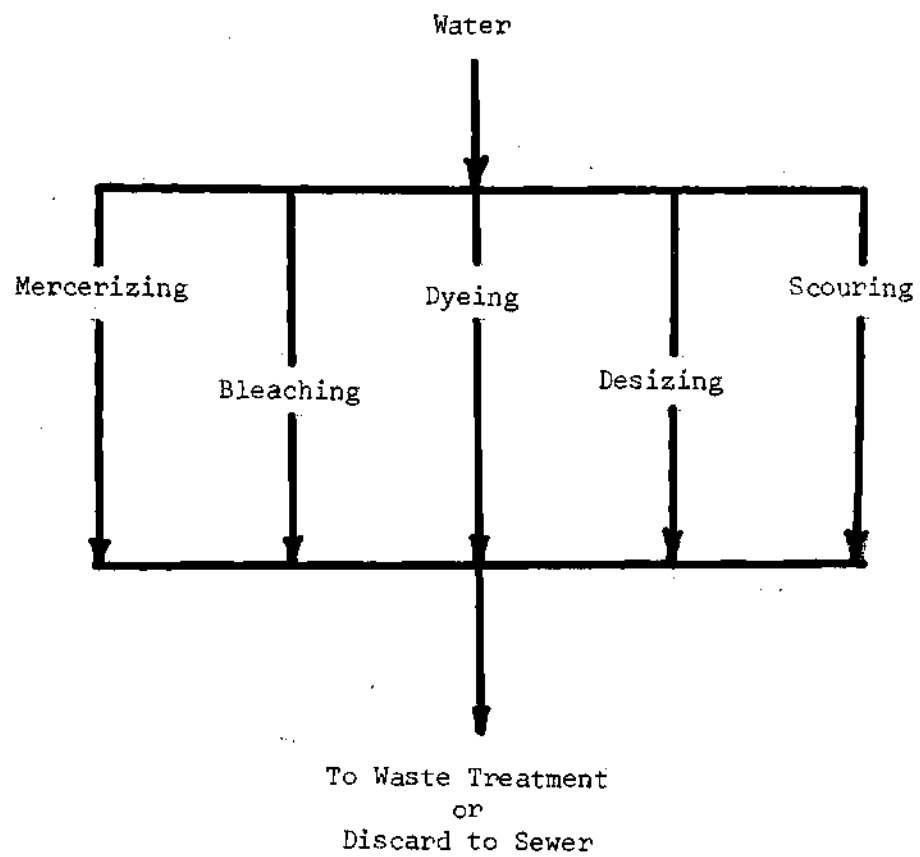


Figure 8. Typical Process Water Flow

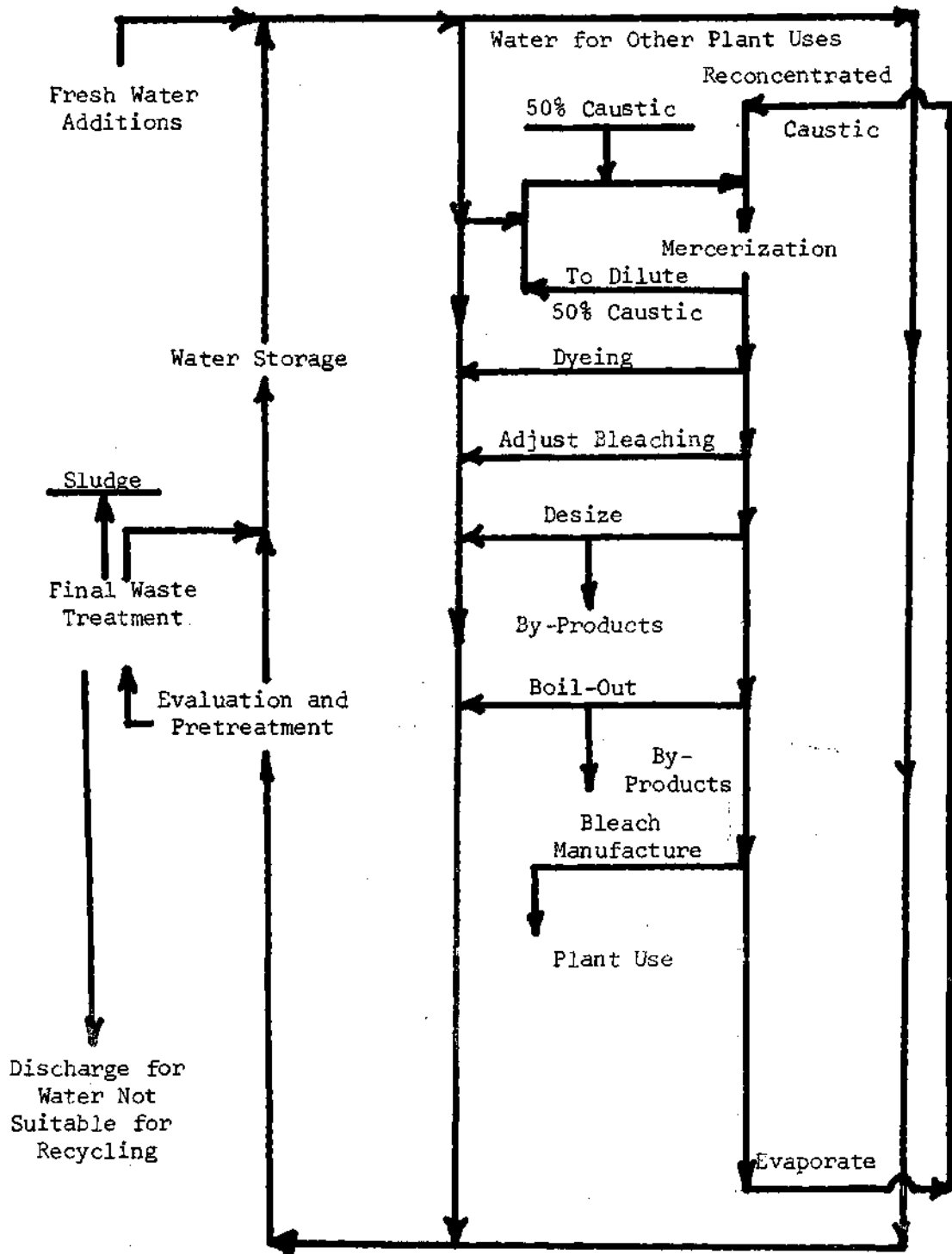


Figure 9. Proposed Flow Diagram

him to improve the competitive standing of his firm.

#### Recommendations

Unfortunately, all progress in the direction of this ideal system is not simple and straightforward application but must be developed through research and modifications to existing systems. There are many problems inherent with the concept of recycling of plant effluent. Here, the textile finisher may have one distinct advantage in that the water can be given any practical attributes through treatment and without contending with the introduction of outside liquid from a polluted stream.

It should be recalled that this research was an investigation into the concept of recovery of a chemical and can be expanded to include the operation of a cotton bleaching, dyeing and finishing plant in a closed system effluent cycle. This general classification embraces five specific areas of investigation contributing to a specific ultimate affirmative conclusion: Cotton bleaching, dyeing and finishing plants can be operated economically with closed, partial or selective recycle of process and plant effluent. The five areas of recommended research are:

- a. An investigation into the problems of using process effluents and treated plant effluent in the make-up of dyebath solutions.
- b. An investigation into the nature of the by-products which may be derived from the effluents of desizing and kier boiling, and the combined plant effluent prior to any waste treatment.
- c. An investigation into the manufacture of hypochlorite bleach from dilute caustic solutions obtained through the recovery of alkaline wash waters from mercerizing.

d. Research into the nature of the waste treatment facilities necessary to classify, segregate, treat and store process effluents to the extent required to render the water obtained satisfactory for general or selected plant process reuse.

e. Evaluation and consolidation of the above completed research into a practical design, operation of the system on a pilot plant scale and final design of a system which could be expanded into commercial practice.

#### Dyebath Solutions

The experienced dyer in the plant may shudder at the thought of using plant waste water to assist in imparting color to fabrics. A fact the manufacturer does not usually consider is that the water used is the same water the plant a few miles upstream has dumped as waste, thus requiring the water to be treated to make it satisfactory for further plant use. This problem exists even though a stream can usually recover promptly from pollution loads. Not only are the industrial users plagued with the necessity for pretreatment, but municipal water purification plants also have to contend with similar problems. The effect of the various process effluents upon the dyeing solutions should be studied with a view to developing a plan for the required degree of treatment to make successful reuse of process or plant effluent for dyeing economically possible. In this investigation, the possibility of reuse of combinations of process wastes to good advantage cannot be overlooked.



### By-Product Recovery

This aspect of consideration for the treatment of textile finishing wastes represents perhaps the ultimate goal toward the development of a plan for dealing with pollution problems by preventive measures. Even though there are numerous examples, recovery of wool grease and dilute sulphite liquor to name two specific cases, where the by-products derived from the waste effluent became too valuable to consider discarding or destroying by waste treatment methods. The area of by-product recovery from integral plant processes is a most neglected aspect of textile processing. Nemerow (46) estimated that some 200,000 tons of cotton impurities containing valuable waxes, pectins and alcohols, are being dumped into the sewers each year. Masselli (47) calculated that, on an annual basis, more than 400,000 tons of glucose can be recovered solely from the desizing waste effluent. Since starch is one of the major pollution problems, the effect of the pollution reduction would be to reduce the BOD load to be treated by 45 to 94 per cent (48). The areas most fertile for the possible isolation of valuable by-products in some quantity are in the processes of desizing, kiering, open boil out and scouring, with bleaching being considered also if done in connection with any of the above processes.

### Bleach Manufacture

The dilute caustic solutions recovered from mercerizing, kiering and scouring operations can theoretically be converted into hypochlorite bleach which can be reused at the plant. As has been pointed out in this research the consideration of such a process appears practical and

was demonstrated in the laboratory with solutions taken from the mercerizer in the mill which cooperated in this study. However, difficulties were noted by the author and additional research is required to refine and place the process on such a scale that it can be accomplished on a continuous basis in the plant. It is the author's opinion that the problems encountered will be those of normal process design with considerable ingenuity required in manipulating the dilute solutions under effective process control.

#### Water Treatment Facility

The water treatment facility required will be essentially a purification process utilizing waste treatment technology. In this area of investigation, the effluent of each process should be studied independently and in relation to other combinations. Process modification within the plant could be considered where necessary to facilitate the treatment of the segregated or combined waste. The facility envisioned would be one such as depicted in Figure 10, with full provision made for classification, separation, storage, remixing, varied treatment possibilities, sludge separation and purified water storage. The scheduling and operation of such a facility cannot be accomplished when viewed as a waste treatment facility but only as an adjunct to or part of the total plant flow process.

#### Recycle System

In this phase, the entire system would be investigated to evaluate and consolidate the findings of the preceding research. In addition, the effluent from each process could be examined for the possibility of waste

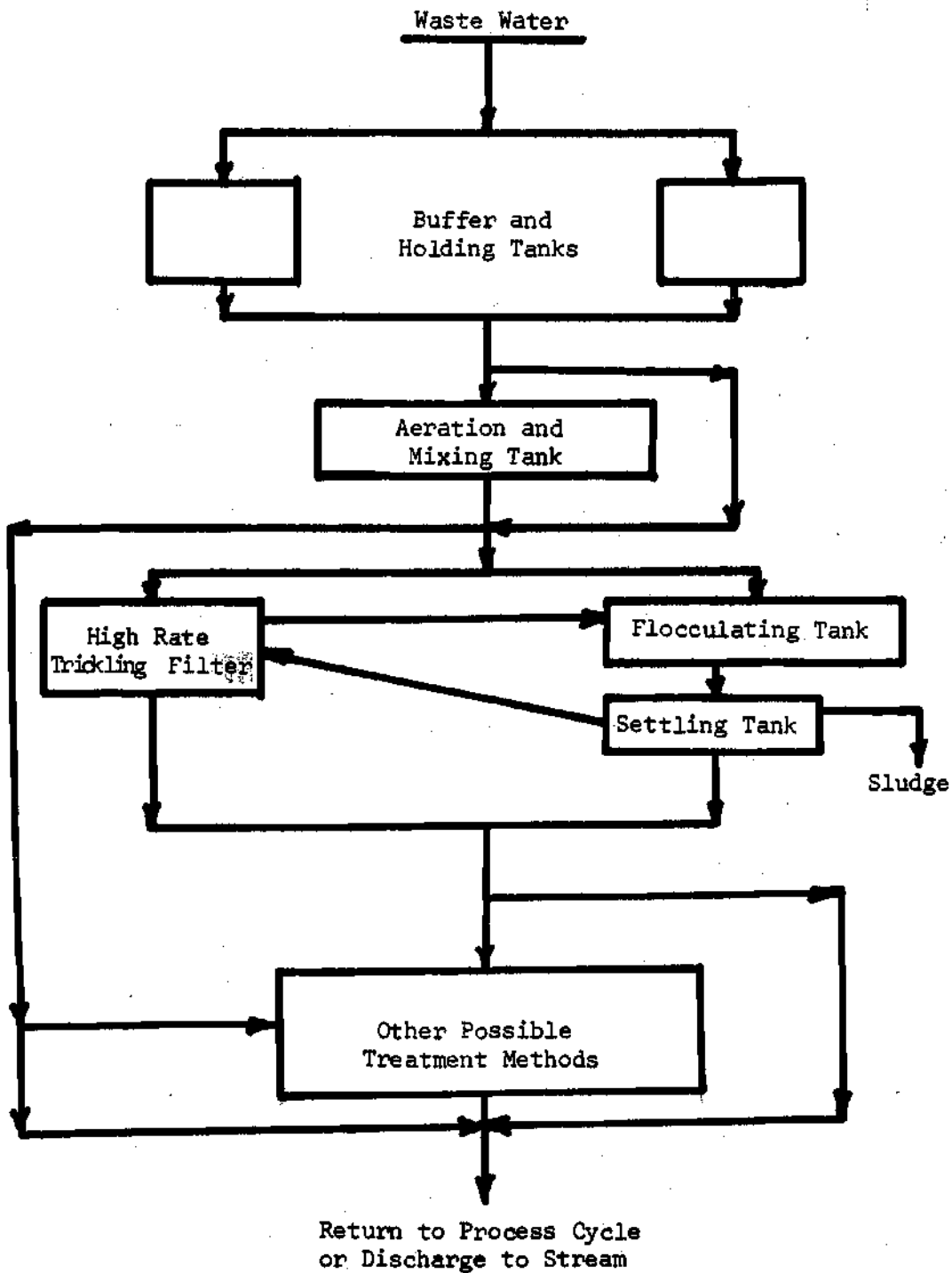


Figure 10. Water Treatment Facility Flow Diagram

heat utilization in other processes through a heat exchange medium. The normal production cycle of the plant could be studied in this phase for the profitable shifting of peak production loads in relation to the effluent cycle. In the case of a specific plant, a pilot operation would be set up and the design of a full-scale application compiled from the experience gained. This proposed recycle plan cannot be accomplished through looking at each process as a unit but as part of the total plant flow.

Upon the successful implementation of such a plan, the textile industry will have made a major contribution to the conservation of this nation's natural resources, gained the economic benefits obviously inherent and substantially contributed to the technological advancement of the industry.

APPENDICES

Table 6. Results of Local Survey of Textile  
Waste Treatment Facilities

Plant	Nature of Business	Degree of Treatment
1	Wool Bleaching and Dyeing	Biological, Chemical and By-Product Recovery
2	Cotton Bleachery and Dyehouse	By-Product Recovery-- Discharge to City Sewer
3	Cotton Bleachery and Dyehouse	Settling
4	Cotton Bleachery and Dyehouse	Settling
5	Cotton Bleachery, Dyehouse and Printing Works	Discharge to City Sewer
6	Cotton Bleachery and Dyehouse	Discharge to River
7	Cotton Printing and Finishing	Discharge to River
8	Cotton Raw Stock Dyeing	Discharge to River
9	Coating Tire Fabrics	Biological, Chemical
10	Cotton Bleaching, Dyeing and Printing	Discharge to Stream
11	Cotton Raw Stock and Yarn Dyeing	Discharge to City Sewer

Table 7. Field Observations: January 18, 1965

Sample Point	Time	Temperature (Fahrenheit)	Appearance
Outside Air	2:30	56	
1	2:30	108	Dark green. Clear.
2	2:38	94	Dark green. Turbid.
Tap Water	2:55	49	

Table 8. Field Observations: January 25, 1965

Sample Point	Time	Temperature (Fahrenheit)	Appearance
Outside Air	2:55	60	
1	2:55	110	Dark green. Clear.
	2:57	108	Dark green. Clear.
2	3:00	98	Dark green. Turbid.
3	3:15	89	Dark green. Turbid.

Table 9. Observations and Measurements: April 9, 1965

Sample Point	F I E L D			LABORATORY	
	Time	Temperature (Fahrenheit)	Appearance	Temperature (Centigrade)	pH
Outside Air	11:30	82			
1	11:30	110	Dark green. Clear.	34	11.4
1	11:38	110	Blue green. Clear.	34	4.3
2	11:50	102	Dark green. Turbid.	30	11.2
3	12:15	96	Dark green. Turbid.	30	11.4
4	12:25	68	Colorless.		

Table 10. Observations and Measurements: April 28, 1965

Sample Point	F I E L D			LABORATORY		
	Time	Temp. (°F.)	Appearance	Temp. (°C.)	pH	Specific Gravity
Outside Air	1:00	78				
1	1:00	110	Dark green. Clear.	36	11.6	
2	1:10	100	Dark green. Turbid.	34	11.6	
3	1:30	92	Dark green. Turbid.	30	11.8	
4	1:22	67	Colorless.			
Wash Box 1	1:45	128	Colorless.	36	12.4	1.022
1	1:58	126	Colorless.	36	12.3	1.022
2	2:00	134	Colorless.	42	12.0	1.008



Table 11. Observations and Measurements: May 5, 1965

Sample Point	F I E L D			LABORATORY		
	Time	Temp. (°F.)	Appearance	Temp. (°C.)	pH	Specific Gravity
Outside Air	1:25	83				
1	1:25	105	Dark Greenish. Clear.	36	11.6	1.000
1	1:30	105	Bluish. Clear.	36	11.8	1.000
2	1:40	100	Dark Greenish. Turbid.	32	11.9	1.000
3	1:52	92	Dark Greenish. Turbid.	32	11.9	1.000
	2:03	66	Colorless.			

Table 12. Observations and Measurements: May 13, 1965

Sample Point	F I E L D			LABORATORY	
	Time	Temperature (Fahrenheit)	Appearance	Temperature (Centigrade)	pH
Outside Air	3:40	88			
1	3:40	110	Light Bluish. Foamy.	38	10.4
1	3:48	110	Light Greenish. Foamy.	36	10.9
2	3:55	102	Dark Greenish. Turbid.	36	11.7
3	4:00	99	Dark Greenish. Turbid.	35	11.5
4	4:08	65	Colorless.		

Table 13. Observations and Measurements: June 2, 1965

Sample Point	FIELD			LABORATORY		
	Time	Temperature (Fahrenheit)	Appearance	Temp. (°C.)	pH	Specific Gravity
Outside Air	2:15	92				
1	2:15	118	Dark Bluish. Clear.	40	11.7	
1	2:22	117	Bluish. Clear.	38	11.5	
2	2:30	119	Dark Greenish. Turbid.	37	11.6	
4	2:30	99	Dark Greenish. Turbid.	35	11.5	
Wash Box 1		158	Clear.	42	11.4	1.034

Table 14. Record of Sample Distillation:  
Carbon Tetrachloride Solvent  
(May 1, 1965)

Time	Fraction Collected	Maximum Temperature (Centigrade)	Reflux (Drops per Second)	Volume Collected (ml.)	Heating Unit Setting (Degrees Centigrade)
11:15		29			65.0
11:21	First Drop	44	1		65.0
11:21	1	54	2	10	67.5
11:31	2	54	2	10	70.0
11:41	3	52	3	10	72.5
11:55	4	53	2	10	75.0
12:04	5	59	2	10	80.0
12:12	6	60	2	10	82.5
12:12	7			18	82.5

Table 15. Record of Sample Distillation:  
Carbon Tetrachloride Solvent  
(May 8, 1965)

Time	Fraction Collected	Maximum Temperature (Centigrade)	Reflux (Drops per Second)	Volume Collected (ml.)	Heating Unit Setting (Degrees Centigrade)
9:45		32			75.0
9:51	First Drop	44	1		75.0
9:51	1	54	2	10	75.0
9:53	2	56	2	10	75.0
9:54	3	56	2	10	75.0
9:56	4	54	3	10	75.0
9:59	5	55	3	10	75.0
10:00	6	58	2	10	75.0
10:02	7	59	2	10	75.0
10:04	8	60	2	10	75.0
10:08	9	61	2	10	76.0
10:08	10			23	76.0

Table 16. Record of Sample Distillation:  
Carbon Disulfide Solvent  
(May 11, 1965)

Time	Fraction Collected	Maximum Temperature (Centigrade)	Reflux (Drops per Second)	Volume Collected (ml.)	Heating Unit Setting (Degrees Centigrade)
2:10		30.0			55
2:32	First Drop	38.0	1		55
2:41	1	45.0	2	10	58
2:46	2	45.0	2	10	60
2:51	3	45.0	2	10	63
2:55	4	45.0	2	10	64
3:00	5	45.5	2	10	64
3:05	6	45.5	2	10	64
3:09	7	45.5	2	10	64
3:14	8	45.5	2	10	64
3:19	9	46.0	2	10	64
3:19	10			18	64

Table 17. Record of Sample Distillation:  
Carbon Tetrachloride Solvent  
(June 3, 1965)

Time	Fraction Collected	Maximum Temperature (Centigrade)	Reflux (Drops per Second)	Volume Collected (ml.)	Heating Unit Setting (Degrees Centigrade)
1:12		32			75.0
1:18	First Drop	45	1		75.0
1:24	1	56	2	20	75.0
1:30	2	60	2	20	75.0
1:34	3	64	2	20	75.0
1:39	4	64	2	20	75.0
1:47	5	60	3	20	75.0
1:52	6	63	3	20	75.0
1:57	7	64	2	20	76.0
2:01	8	64	2	20	76.0
2:06	9	66	2	20	77.0
2:06	10			25	77.0

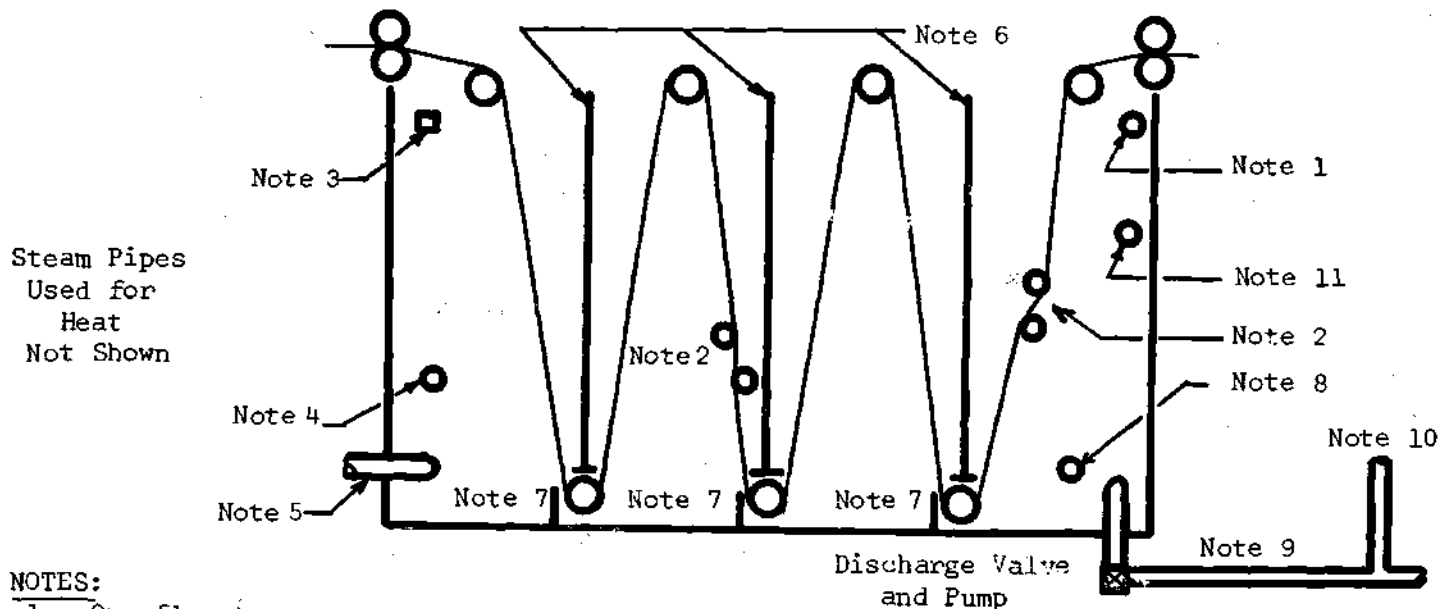
Table 18. Record of Sample Distillation:  
Carbon Disulfide Solvent  
(June 3, 1965)

Time	Fraction Collected	Maximum Temperature (Centigrade)	Reflux (Drops per Second)	Volume Collected (ml.)	Heating Unit Setting (Degrees Centigrade)
1:15		32.0			57
1:46	First Drop	39.0	1		57
1:54	1	45.0	2	2	60
2:05	2	45.0	2	20	60
2:18	3	45.0	1	20	60
2:34	4	45.0	1	20	62
2:43	5	45.5	2	20	64
2:55	6	45.5	2	20	64
3:04	7	45.5	2	20	64
3:13	8	46.0	2	20	66
3:20	9	46.0	2	20	66
3:20	10			27	66

Table 19. Water Price Scale Used  
in Water Value Computations

	Cubic Feet	Per 100 Cubic Feet
First	100	\$1.57
Next	100	.37
Next	100	.29
Next	100	.24
Next	10,000	.22
Next	100,000	.19
Next	100,000	.17
Next	100,000	.15
All Over		.11

NOTE: 7.481 Gallons of water equals 1 cubic foot.  
0.134 Cubic feet of water equals 1 gallon.

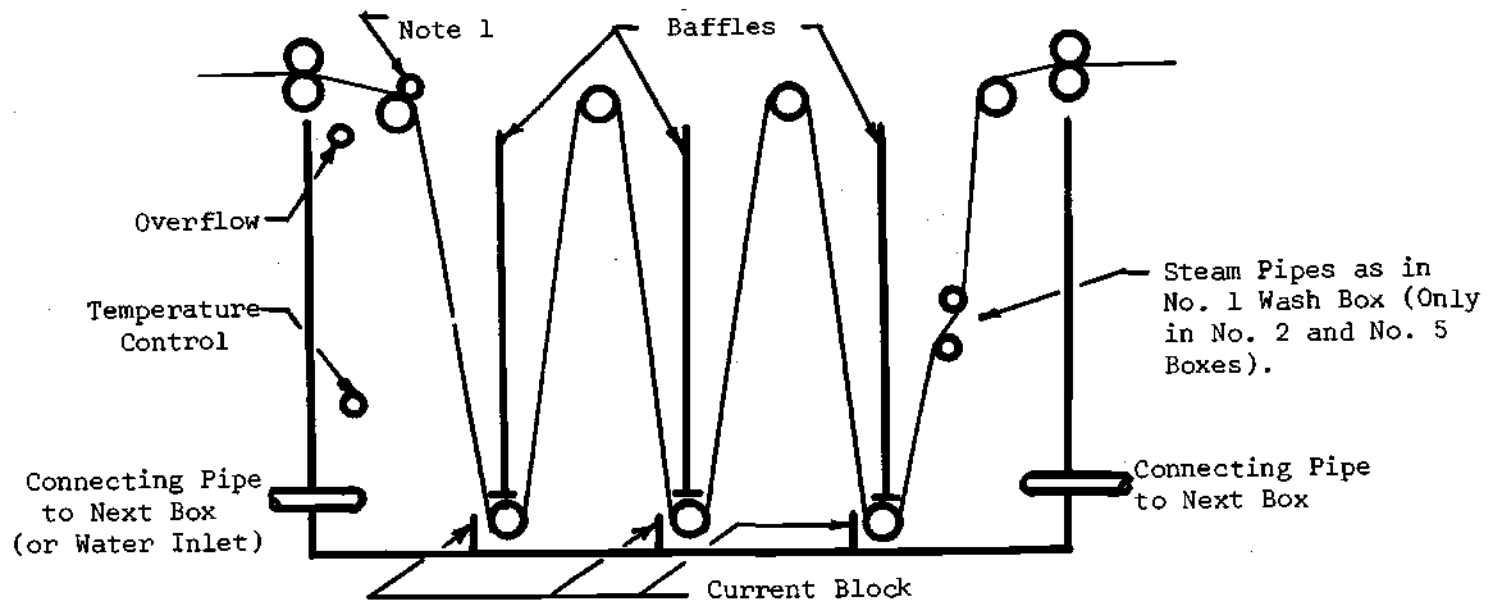


**NOTES:**

1. Overflow to sewer.
2. Steam pipes receiving continuous supply of steam.
3. Device to measure level of water and actuate inlet valve on No. 5 wash box.
4. Temperature sensor controlling steam for heat.
5. Connecting pipe between boxes.
6. Removable baffles. Bottom of baffle mounted as close to top of roll as practical.
7. Current block mounted as close to roll as practical.
8. Electrode to measure concentration of electrolyte. Controls discharge rate.
9. Discharge to storage tank. Routed through heat exchanger. Overflow to sewer.
10. Spray to tenter frame.
11. Drain from drip pan on tenter frame.

Figure 11. Design of First Wash Box

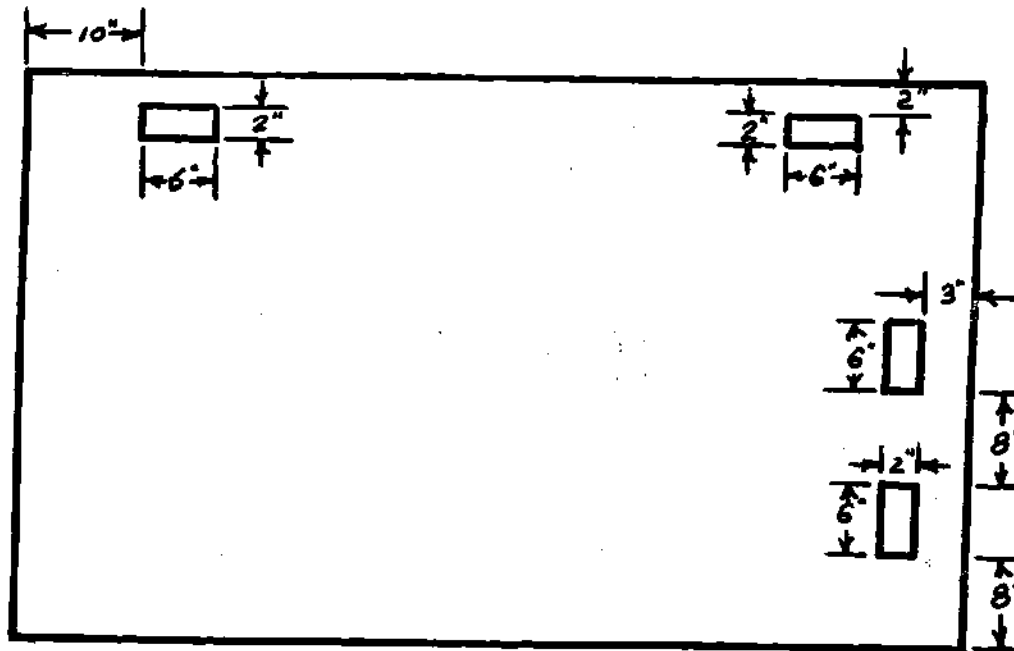




NOTES:

1. Potentiometer (only on No. 5) to sense electrolyte content of fabric and regulate production speed.
2. Steam pipes used for steam heat not shown.

Figure 12. Design of Typical Wash Box



Note: Baffle to be cut to inside dimensions of wash box.  
 Three required per wash box.  
 Material should be durable.  
 To be mounted in brackets secured to sides of wash box.

Figure 13. Wash Box Baffle Design

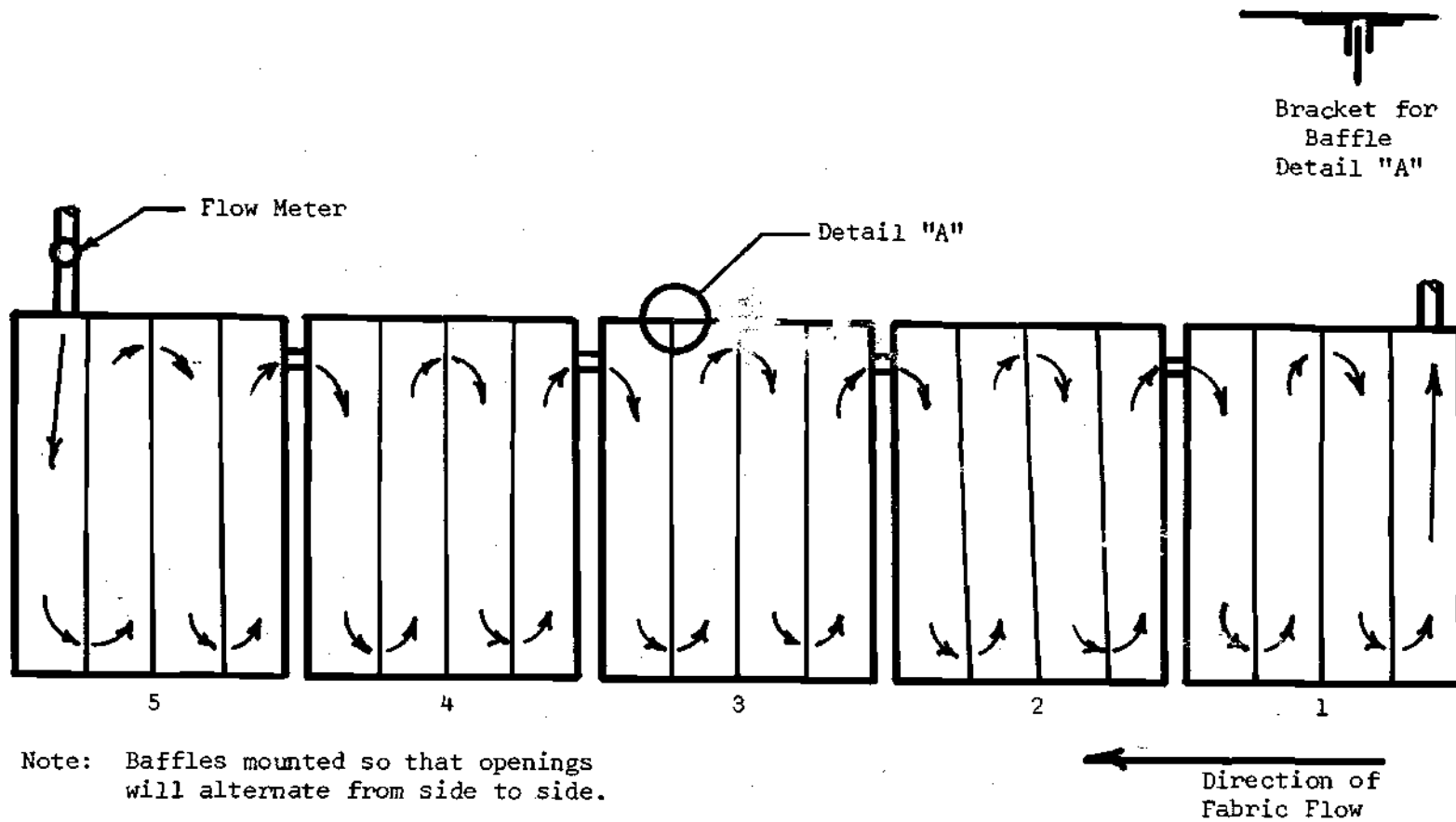


Figure 14. Washing System Flow Diagram

Useful Formulae:

$$\text{Baume}^{\circ} = 145 - \frac{145}{\text{Sp. Gr.}}$$

$$\text{Sp. Gr.} \times 1000 = (5 \times \text{Twaddell}) + 1000$$

$$\text{Sp. Gr.} = \frac{145}{145 - \text{Be}^{\circ}}$$

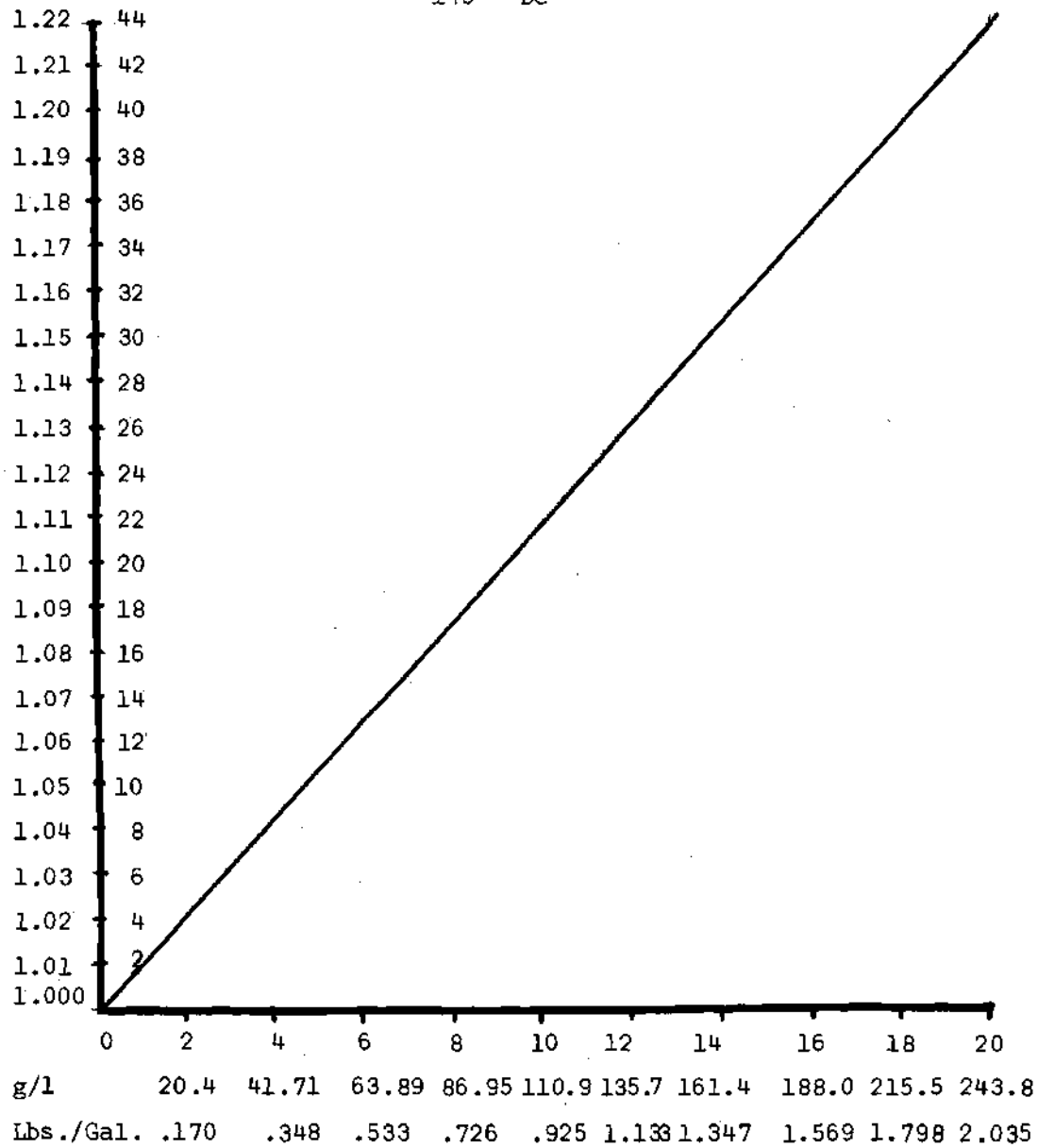


Figure 15. Physical Attributes of Caustic Soda in Solution

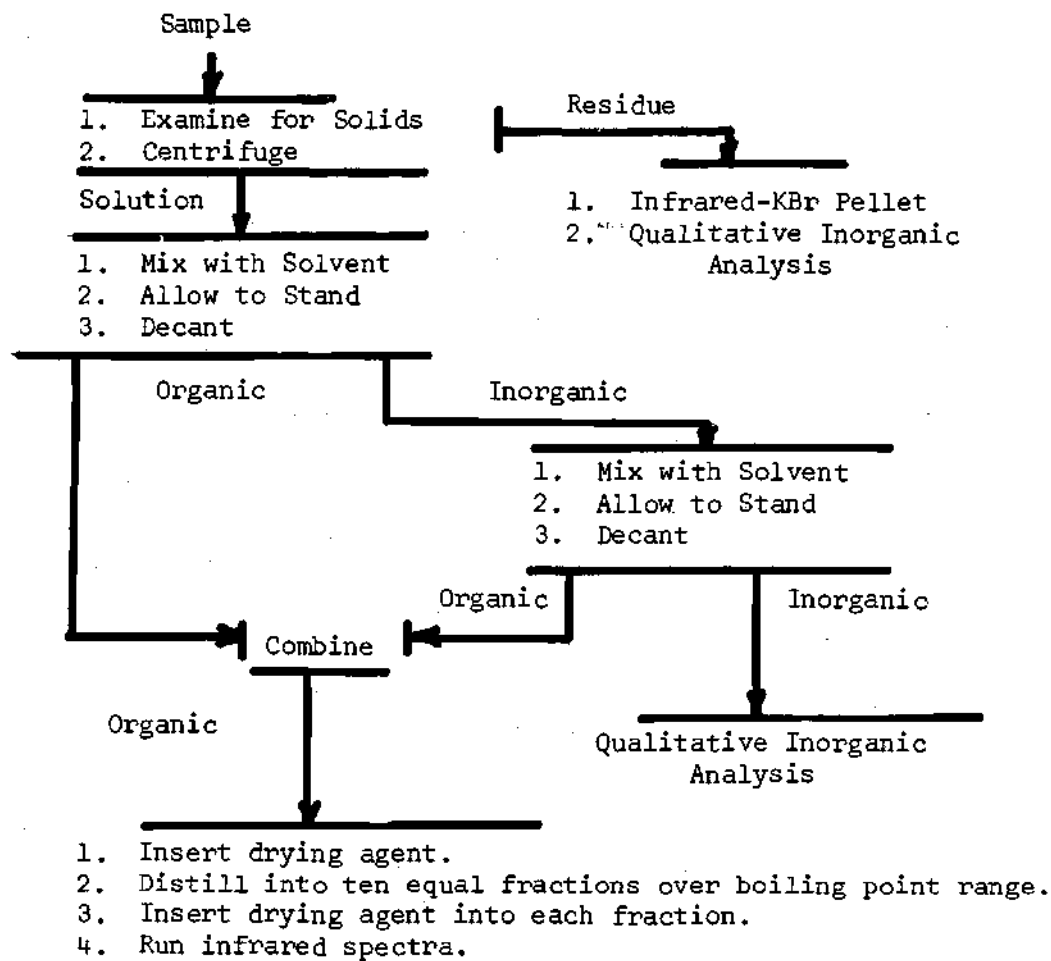


Figure 16. Design of Sample Analysis Program to Check for Impurities in Mercerization Wash Solutions

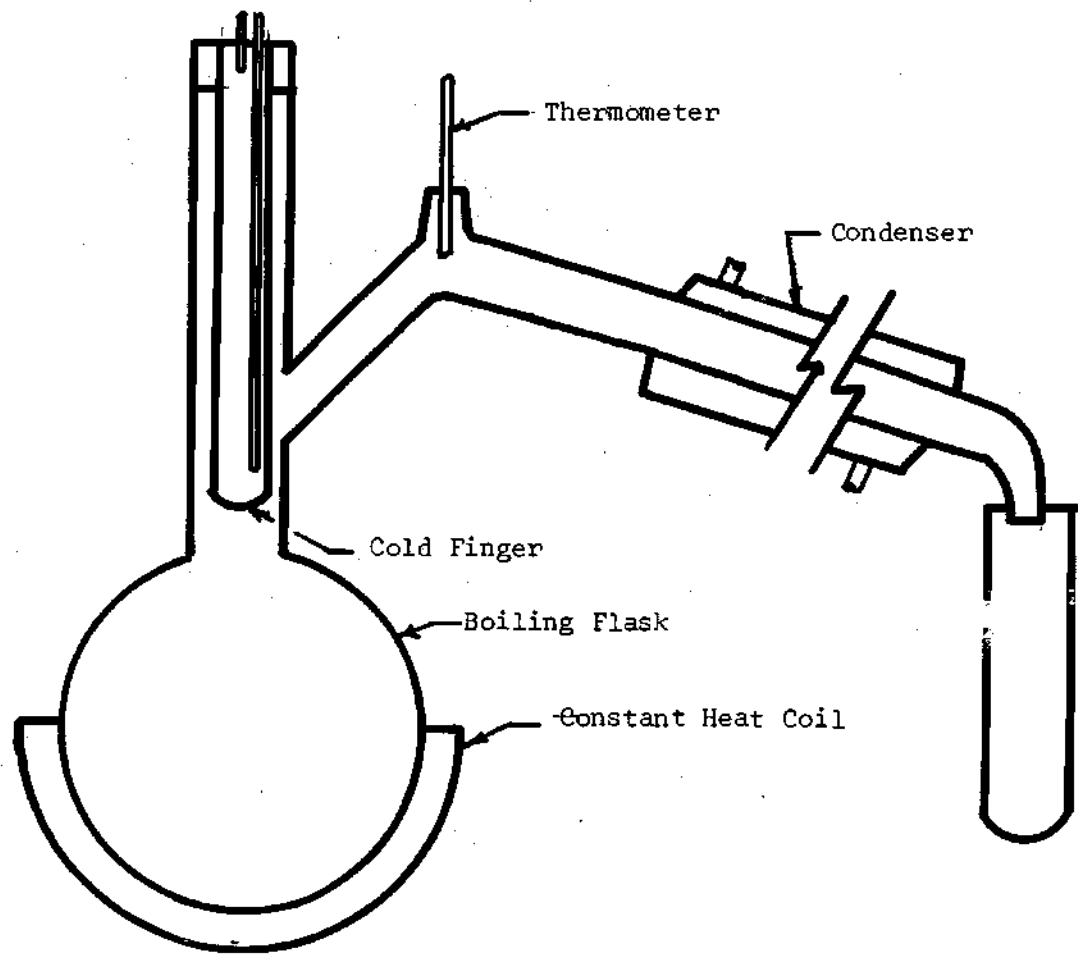


Figure 17. Diagram of Distillation Assembly

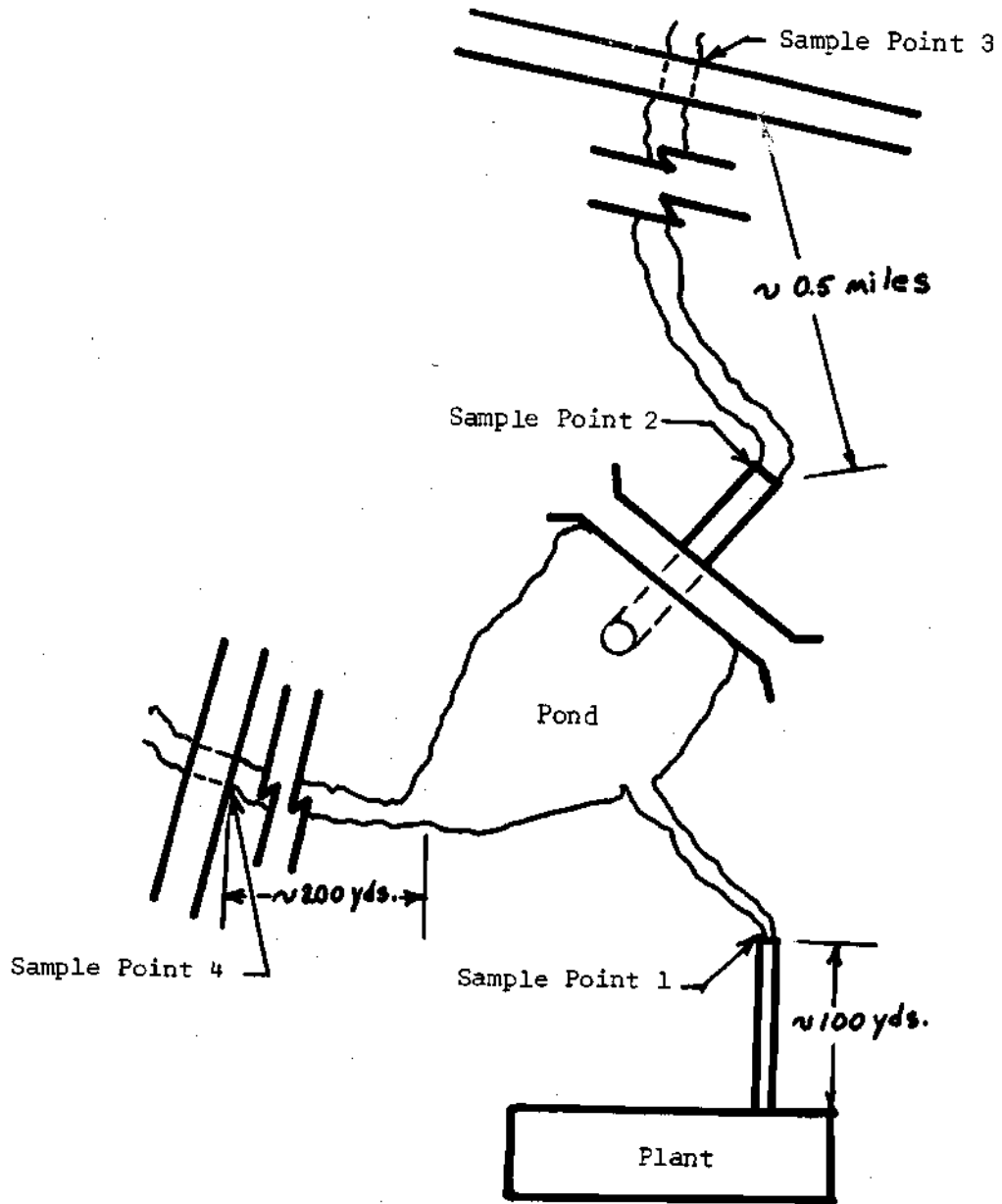


Figure 18. Location of Sampling Points  
in Relation to Plant

Table 20. Condensed Information from Infrared Absorption Spectra of Mercerizer Wash Solutions

Spectrum Number	Wave Length (Microns)															Solvent	Thickness of Cell (MM)
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15		
1			W			M		W								CCl <sub>4</sub>	0.5
2			M	M		M		W		W						CCl <sub>4</sub>	0.5
3			M	M		M		W		W						CCl <sub>4</sub>	0.5
4			M	M		M		W		W						CCl <sub>4</sub>	0.5
5			M			M	M	W		W						CCl <sub>4</sub>	0.5
6			M			M		W		W						CCl <sub>4</sub>	0.5
7			M	M		M		W		W						CCl <sub>4</sub>	0.1
8			M	M		M		W		W						CCl <sub>4</sub>	0.1
9			M	M		M		W		W						CCl <sub>4</sub>	0.1
10			M	M		M		W		W						CCl <sub>4</sub>	0.1
11			M	M		M		W		W						CCl <sub>4</sub>	0.1
12			M	M		M		W		W						CCl <sub>4</sub>	0.1
13			M	M		M		W		W						CCl <sub>4</sub>	0.1
14			M	M		M		W		W						CCl <sub>4</sub>	0.1
15			M	M		M		W		W						CCl <sub>4</sub>	0.1
16			M	M		M		W		W						CCl <sub>4</sub>	0.1
			M	M		M		W		W						CS <sub>2</sub>	0.1

NOTE: W = weak; M = Medium; V = Strong; B = Broad; S = Sharp; P = Multiple Peaks.



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