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Membrane Filtration Techniques Used for Recovery of Dyes, Chemicals and Energy

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When we consider wastewater, energy and chemical recycle in the textile industry, it is important to first look at the impact the industry makes on the national economy and determine what effect recycling will have on the available resources. For this evaluation the data in Fig. 1 shows the quantity of fibers, water and energy consumed on a daily basis. The cost of water is estimated to be $0.5 million per day, waste treatment $1 million per day and energy $5 million per day. An additional $30 to $40 million is calculated for the fibers used and the total nears $50 million per day. The total also includes dyes, finishes and other chemicals. This is truly an impressive number for an industry that was initiated in the Garden of Eden.

ABSTRACT

The continued increase in the cost of chemicals, energy and water makes their recovery more important today than it was 20 years ago when ultrafiltration and hyperfiltration were first introduced to the textile industry. While the filtration techniques have been used at only a few installations, these plants have been able to save enough to pay for the recovery process in one to two years. One key to having a successful recovery operation is to have good automatic control of the process. This can drastically improve the economics of the textile process as well as minimizing the cost of the recovery system. The recovery of PVA, indigo, caustic and preparation chemicals will be presented.

KEY TERMS

Bleaching
Chemicals Recovery
Dyeing
Dyes Recovery
Energy Conservation
Finishing
Membrane Filtration
Preparation
Ultrafiltration
Wastewater Treatment

The concept of recycle of wastewater has been actively discussed and investigated for the past 20 years in the textile industry. The early application of ultrafiltration to PVA size recovery was accepted and used in a full plant scale installation (*1*) almost 20 years ago. The process was successful then and continues to perform well. One limitation of the use of ultrafiltration was diversity of sizing chemicals that are in use. Controlling the size used on greige fabric is also necessary if the plant wants to recover PVA size from its preparation range. In spite of these limitations, there has been an increase in the number of plants that are recovering PVA size finish. While recovery of size is only done in a small fraction of the finishing plants, interest in the recovery process is strong and the need appears to be increasing (*2*).

As hazardous waste regulations increase and restrictions on the discharge of trace metals increase, the textile industry will be faced with the continual upgrading of its treatment facilities (*3*). The increase in the cost of solid waste disposal and the effect this will have on sludge disposal is illustrated in Fig. 2 (*4*). While this is not directly connected to the recycle process, it illustrates the continued increases in the cost of all aspects of waste treatment.

In addition to the cost of solid waste disposal, the cost of energy is much higher today than it was almost 20 years ago when the recovery of PVA was first introduced. Since all of these factors continue to place pressure on the industry to eliminate waste discharges, several processes where recycling may be applied or is being applied with considerable success will be examined.

PVA Recovery

Recovery of PVA was one of the first recycle processes to be used by the textile industry. The factors that determine the cost are shown in Fig. 3. The recovery from one range can amount to over $5600 per day. The equipment can be paid for in approximately one to two years. This accounts for the strong interest in the recovery of PVA today. A diagram of a PVA recovery system is shown in Fig. 4 (*5*). An important factor is that when size is recovered, 60,000 gallons of discharge from each range per day is eliminated from the waste stream. While some blow down or clean up wastes are still discharged, the volumes are small and only contribute a fraction to the unrecycled waste stream. Costs of PVA, fresh water and waste treatment have increased signif-

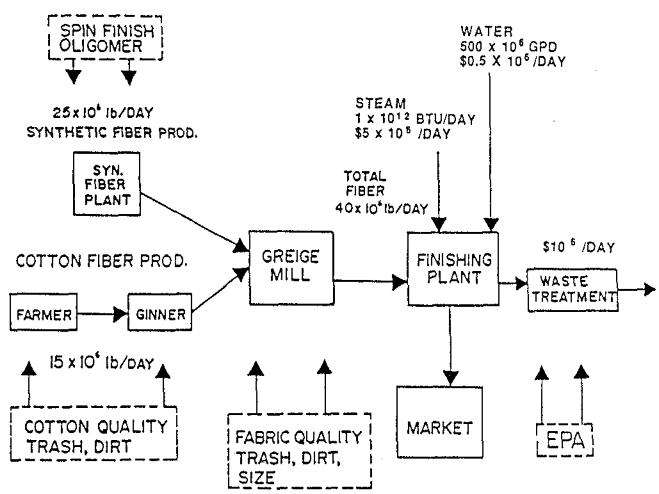


Fig. 1. Factors affecting finishing plant operation.

icantly. Recycling of PVA is more attractive today than it was previously.

Caustic Recovery

The recovery of caustic from the mercerization process is a common practice in the textile industry. Mercerizer rinse water is normally recovered for evaporation when its concentration is above 2-3%. It is discharged to waste treatment when its concentration is below this level. Impurities from the fabric build up in the used caustic solution as caustic is removed. Eventually the solution waste stream must be discharged and the mercerization process requires fresh caustic solution. An alternative to this procedure is to use an ultrafiltration membrane to filter the caustic rinse water before the solution goes to the evaporator. The clarified and concentrated caustic solution is then ready for reuse and the consumption of caustic is significantly decreased.

A diagram of a caustic recovery solution using ultrafiltration is shown in Fig. 5 (*6*). The system shown has been operating for five years and has been increased in size since it was first installed (*7*). After four years of operation, the payback of invested capital was reported to be within 12 to 18 months (*7*) and amounts up to $1.5 million in raw material savings per year. A return on investment such as this makes the process attractive to many plants.

Indigo Recovery

Recovery of indigo dye is an example of a system suited for the ultrafiltration process. The dye has a significant value at $9 per pound and because of its deep blue color would be readily visible in a receiving stream. If it is possible to recover the dye, pollution can be reduced and a savings in resources can be realized. The Liberty Plant in Liberty, S. C., has been recovering indigo since 1981 and paid for the recovery system in less than two years of operation (*8*).

The recovery system, a Dorr-Oliver produced by the Amicon Division of W. R. Grace & Co., uses a vinyl-sulfone membrane. The dyeing process at the Liberty Plant has been modified so that when sulfur dyes are used they are applied after the indigo dye is applied thereby not contaminating the indigo waste stream.

The recovery system is a multistage system with one feed pump and a bleed valve as shown in Fig. 6. Each stage automatically establishes a steady state concentration which becomes progressively higher as the concentration increases. The final stage reaches the maximum concentration of indigo for reuse. To minimize the cost of the membrane system, the indigo waste stream should be produced from countercurrent flow washboxes adjusted to have a total flow nearing 25 gallons per minute. When the countercurrent flow is adjusted as such, it is possible to start recovery with an indigo concentration of near 800 ppm. This reduces the cost of the membrane system by half to a third below that normally found where the flow is 100 gallons per minute and the indigo concentration is 200 ppm.

The system is preceded by a 325-mesh vibrating screen used for lint removal from the waste stream before it enters the multistage membrane system. After the indigo is concentrated it is filtered through a 200-mesh basket strainer and stored in a holding tank capable of holding a four-day supply of concentrate. The ratio of concentration flow to feed flow generally ranges from 1:30 to 1:50. The clear filtrate is

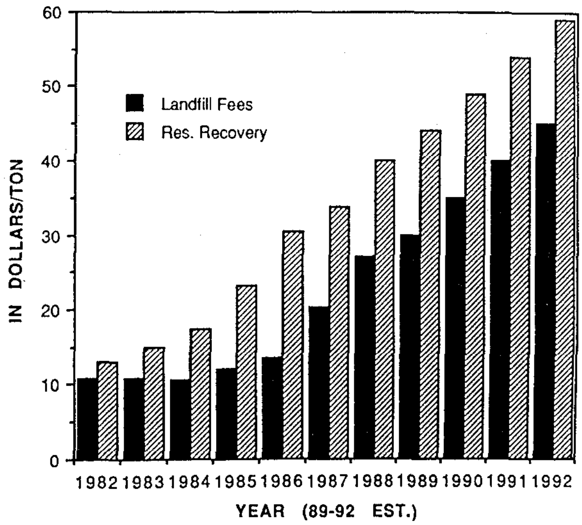


Fig. 2. Tipping fees in the U.S.

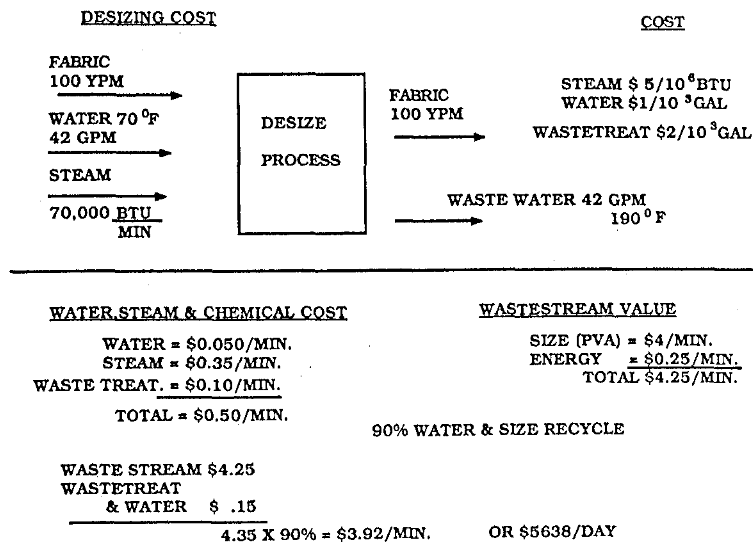


Fig. 3. Value of PVA recovery.

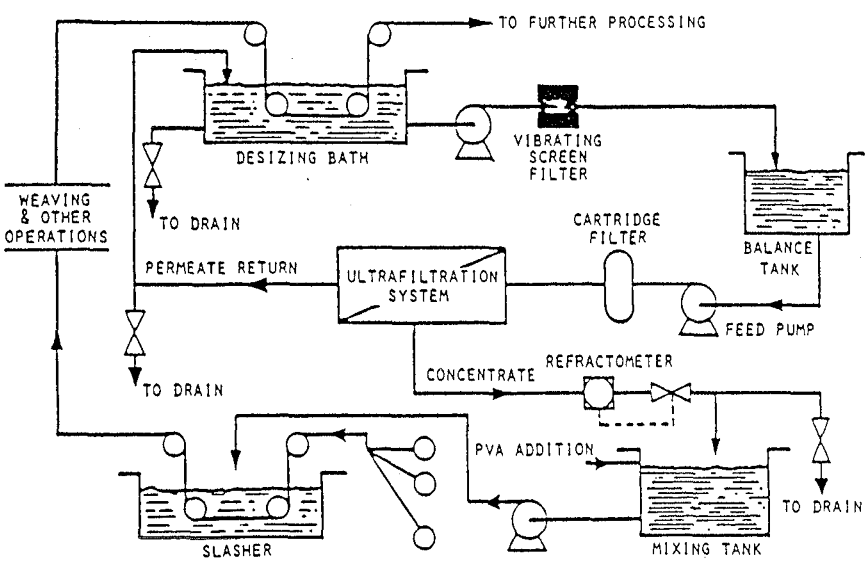


Fig. 4. PVA recovery system.

discharged to waste treatment containing ~~the used chemicals from the dyeing~~ process minus the indigo dye. The indigo recovery system is reported to have paid for itself in less than two years and operates with a minimum of problems.

Preparation

Fabric preparation is the most important step for gaining control of the dyeing and finishing operation. If the fabric is not consistently and uniformly prepared it will be difficult for the dyeing operation to make adjustments to correct for differences in fabric wet pickup. If the subsequent dyeing process is continuous, the problem of preparation nonuniformity will be severe because the fabric will wet out differently or unevenly. If the dyeing process is batchwise, dyeing conditions may have to be changed. All of these factors are well known and the textile industry is well aware of the need for good fabric preparation.

The normal procedure used to attain good preparation is to set the range condi-

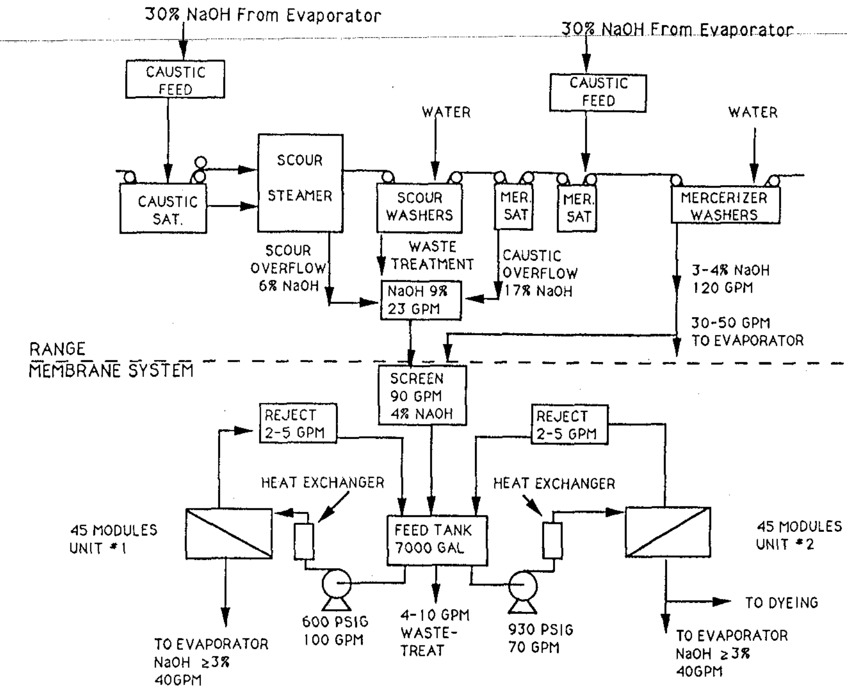


Fig. 5. Caustic recovery system.

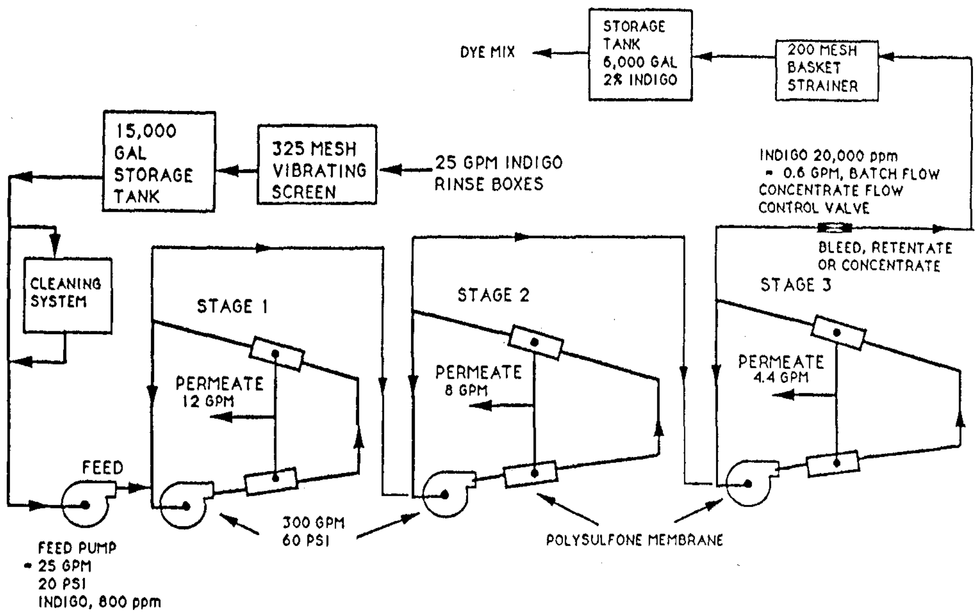


Fig. 6. Indigo range recovery system.

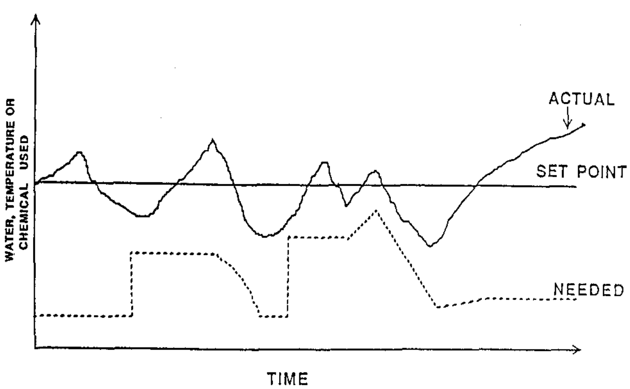


Fig. 7. A process or unit operation without automatic control.

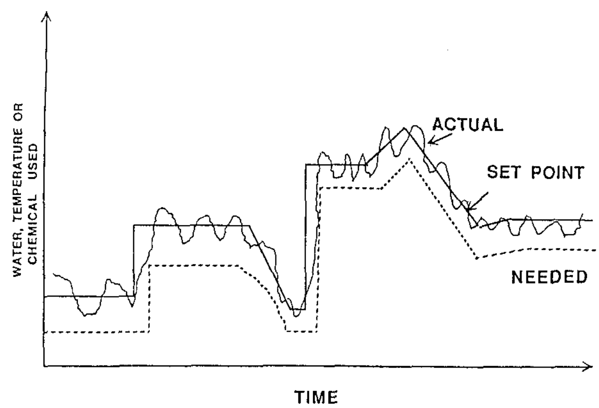


Fig. 8. A process with automated control.

tions to those needed for difficult to prepare fabrics. These conditions are then used for temperature, water flow and chemical feeds and remain at these settings even though they may not be required but a fraction of the time. This is illustrated in Fig. 7. The process illustrated has no automatic process control and stream or water flow will vary as plant water and steam pressure vary. In this regard, fabric requirements may not be met when plant water and steam pressure are low. As shown in Fig. 8, changing to automatic control adjusts supply to need rather than a setting that may have nothing to do with the fabric being prepared at a specific time.

To recover chemicals and hot water from a process it is most important to control the process and know the exact needs for the process. The recovery process can then be designed to an optimum size and the holding capacity necessary for reuse can be designed properly.

Fig. 9 illustrates a preparation range showing three stages: desizing, scouring and bleaching. When the fabric is to be mercerized it must be transferred to a separate range for mercerization. The wash water from mercerization is generally collected for evaporative recovery when it is near 3% NaOH or higher. When it is below 2% it is discharged to waste treatment.

The peroxide washer wastewater may be used directly as feed to the caustic washer. When this is done it is possible to save $120,000 per year. However, the need to pump the water and the retrofit costs have discouraged many textile plants from making the change. The total solids present in the peroxide washwater is generally less than 0.5% and should cause few problems for the caustic washer. One point of caution is the use of silicate stabilizers for the peroxide bleach. The stabilizers could interact with calcium or magnesium salts present in the natural cotton fiber and give a precipitate. In many cases, organic stabilizers are used which do not create a problem. Proper selection of stabilizers can enhance the success of peroxide washwater reuse.

Another source of energy loss in preparation is when the fabric is skyed. Fig. 9 shows two locations where the fabric is skyed. If this practice could be avoided, the cost savings would be $32,000 per year for the range shown.

The caustic washer contains less than 1% total solids. It can be used as a desize washer when the size is not recovered. This is a more difficult option but the potential to save an additional $120,000 makes the process attractive. The decrease in total wastewater flow not only saves on the cost of water, waste treatment and energy but can improve the biological treatment process used by most textile plants. When the plant recycles water, the water flow going to the waste treatment plant is reduced and the retention time available for biological treatment increased. This improves the biological waste treatment if no change is made in the volumetric capacity of the waste treatment system.

The overall potential for savings in caustic, water, steam and waste treatment could be over $300,000 per year. Once demonstrated as practical, installation of a few pumps and one or two screen filters are the only requirements. When sensitive fabrics are processed, the range could automatically adjust flows to meet fabric requirements.

Beck, Beam or Jet Dyeing

The wastewater from dyeing is more difficult to recycle than water from the previously discussed processes. This is because the color of the dyeing wastewater continually changes. An exception to this is the indigo process where the color is always the same. If sulfur dyes are not used of if they are applied after the indigo dye, no contamination occurs in the indigo wastewater. With most piece dyeing operations, the target shades vary drastically. For this reason, it is difficult to separate the recovered dye in sufficient quantities to make the dye reuse practical. Dye recycling has been demonstrated as a successful recovery method (*9*). The major limitation to the practical use of dye recycling as a cost effective recovery method is that the quantity of dye recycled must be sufficient to make it worthwhile.

A materials balance for a dyeing machine is shown in Fig. 10. The water will not always be heated but most of the process steps of concern for potential energy savings operate faster and more effectively with hot water. At the flow rates shown, it is possible to recover over $100,000 per year from energy and water savings. In this case a membrane must be used to remove soluble dyes and must withstand the temperatures of the wastewater which can approach 200F. Pres-

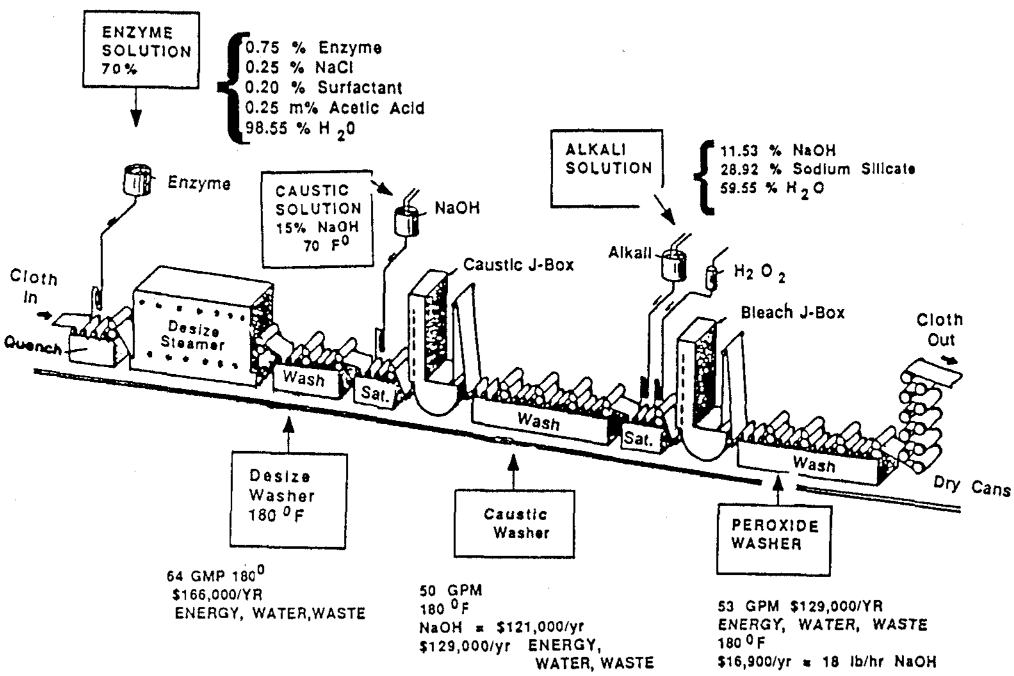
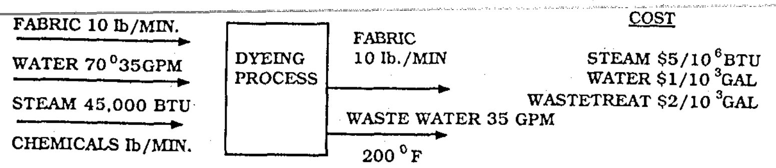


Fig. 9. Preparation range.



WATER,STEAM & CHEMICAL COST

WATER = \$0.04/MIN
STEAM = \$0.23/MIN
WASTE TREAT= \$0.07/MIN
CHEMICALS =\$1.00/MIN

TOTAL \$1.34/MIN

WASTESTREAM VALUE

ENERGY = \$0.19/MIN
CHEMICALS = \$?

TOTAL \$0.19

90% WATER & ENERGY RECYCLE

$$[\$0.11 + 0.19] \times .9 = \$0.27/\text{MIN} \times 1440 \frac{\text{MIN}}{\text{DAY}} = \$388/\text{DAY}$$

\$388/DAY x 300 DAYS/YR. = \$116,000/YR

\$388/DAY x 300 DAYS/YR. = \$116,000/YR

Fig. 10. Three-stage rapid bleach range.

ently, the only membrane capable of handling the conditions is the CARRE manufactured by Du Pont. The membrane has been used successfully to recover dyes and hot water from the beck dyeing process (*9*).

Continuous Dyeing

Recovery of dyes and hot water from the continuous dyeing process was illustrated with the recovery of indigo. The indigo process operates continuously to dye warp yarns which are woven into denim fabric. When dyes and auxiliary chemicals are used to dye large quantities of fabrics, the dyes can be recovered and reused. When the quantity of dye used is small, the recovery of dye is not practical. However, in the case evaluated on a continuous dye range (*10*) the hot water (>180F) and waste treatment savings amounted to \$200,000 to \$300,000 annually depending on the price of oil and the waste treatment process. At this savings a recovery system could pay for itself in one to two years.

Summary

The recovery of chemicals, energy and water for reuse is more economical today than it was 20 years ago when interest was first directed toward the recovery concept. Fuel, water and waste treatment costs are much higher today and the regulations governing wastewater discharge are much more demanding. As fuel costs and wastewater discharge regulations increase, recovery processes become more attractive.

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Yarn Dyeing: Problems and Solutions

Evolutionary changes in yarn dyeing processes were the focus of an AATCC symposium at Charlotte in early April. Based on a survey which asked dyers to suggest the topics they most wanted to hear discussed, the program offered practical approaches to some of the most common problems faced by yarn dyers. Developed by AATCC's Committee on Yarn Dyeing Technology, the program provided registrants the opportunity to take a direct part in the proceedings with questions and suggestions from the floor following each guest speaker and at a concluding series of sessions grouped according to the end-use market served. The following summaries are from the papers that were presented. The group discussions were not transcribed.

Machinery

Optimum Yarn Dyehouse

O. John Harry of Grover Industries told what he would do if he were given the opportunity to build the optimum yarn dyehouse. He said he would build the dyehouse with the flexibility to expand to take advantage of newer equipment or be easily renovated for new processes which may be installed. He also said that dyeing between 150,000 and 200,000 pounds of yarn a week would be large enough to be a profitable operation yet small enough not to require too many layers of management. To make it possible to dye the quantities of yarn most economically, Harry recommended the installation of the larger diameter kier size with packages six or seven high. He feels that the quality produced by this machine is slightly better than most of the 34-inch kiers with packages stacked nine or ten high. He also said the capacity of the dyehouse could be best handled by installing two machines in the 100 to 150 pound size, two machines in the 400 to 500 pound size, four machines in the 1000 to 1500 pound size and one machine in the 2000 to 3000 pound size. In his experience, Harry found that smaller dyeing machines are not profitable and that if the number of smaller dyeing machines is limited, customers are willing to buy larger lots to get a quicker delivery.

Other features Harry would design into the optimum dyehouse include individual microprocessor control of dyeing machine conditions as well as a not water system heated by waste heat recovery and by hot water generated from high pressure dryers. Harry believes that individual dyeing machine control with microprocessors is imperative to a successful operation. The installation of a hot water system in the early stages of construction is easy to justify in terms of long term flexibility and growth.

In Harry's opinion the dye tube winding department has the most important function in the dyehouse. Precision winding equipment should be used because it provides a more uniform flow through the package during dyeing than conventionally wound packages. It also makes it possible to increase the weight of a package without increasing its volume. According to Harry, it is more economical to dye larger loads by increasing the weight of the package.

Assessment of Dye Tubes

Selection criteria for yarn dye tubes was the focus of Peter J. Horn of Ciba-Geigy. A history of the development of the variety of dye tubes and the rationale for selecting one type over another were also discussed. According to Horn, the ideal dye tube does not exist. Selection is always a compromise.

Basic requirements for dye tubes include consideration of the expected performance in dyeing and drying as well as production steps prior to and subsequent to the coloration process. Many automation systems will also limit the range of dye tube selection. The winding ratio—that is the ratio of the inside tube diameter to the outside package diameter—is a key parameter both in dyeing and drying. Liquor flow through a package influences the color uniformity of the yarn.

Materials used to produce dye tubes offer advantages in cost and durability. Polypropylene usage increased about 10 years ago when its quality improved and its durability was increased with glass fiber reinforcement. Stainless steel is still used in the manufacture of dye tubes and is required if the finishing process includes chlorite bleaching. Disposal of either dye tube material is a problem to the package dyer. Horn noted that some European suppliers of polypropylene dye tubes offer a chipper with the sale of the product so that recycling is easier.

The standard conical dye tube introduced in 1937 is still the most popular tube in Europe. It was not until the 1970's that manufacturers of dyeing equipment realized that up to 70% of the total liquor flow could be lost between dye tubes and spacers. Efficient machine loading and flow geometry are critical in producing an evenly dyed package.

Press dye tubes developed to satisfy these concerns as well as answer the need for continuous processing of larger packages, eliminate the need for backwinding and be compatible with newer spinning and weaving equipment. A press dye tube system offers the advantage of leakfree columns with radial liquor circulation, the elimination of spacers, a protected reserve tail for continuous processing and a 40% increase in capacity utilization over standard conical systems. When press dye tubes are made from polypropylene, they are limited to single use. Telescopic dye tube systems combine the advantages of a press system and multiple use of dye tubes.

A continuing trend in dye tubes will be a decrease in the use of conical shapes and stainless steel while improvements in the manufacture of the newer types will accommodate continuous processing and level dyeing of filament yarns. According to Horn, selection of the proper dye tube is a matter of technical argument, cost and ecological conscience.

Automated Materials Handling

Cyrus Wang of Burlington Industries discussed benefits derived from an automated materials handling system in a plant that handles packages of yarn. Package parameters such as weight of incoming shipment and time in inventory were tracked by the system to assist in production planning. An automated storage retrieval system reduces the amount of manual handling required to run the plant and eliminates much of the paperwork traditionally required when yarn is removed from storage, processed in the plant and shipped to the customer. By tracking the product from the entrance to exit of the plant, the user maintains process control.

Horizontally Configured Equipment

For two years Amital Spinning Corp. of New Bern, N.C., has operated a package dyehouse with horizontal package dyeing equipment designed by the Italian manufacturer OBEM. Brenda S. Waters of Amital showed a video of the equipment in operation for dyeing acrylic yarn. According to Waters, Amital has been successful with no-add dyeing and has suffered very few redyes. An overhead crank, remote control unloading, hot water re-use and