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"ELVANOL® POLYVINYL ALCOHOL WARP SIZING RECOVERY"
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Feel free to share this with customers as appropriate.

GDR
INTRODUCTION

Environmental issues are becoming more important to businesses worldwide. Not only are environmental regulations becoming more restrictive, the public is putting increasing pressure on industry to provide products that are environmentally friendly. This applies not only to the products, but also to the processes used to manufacture these products.

At the same time, the global economy is forcing textile manufacturers to face an unprecedented level of competition on a world-wide scale.

To satisfy these demands, chemical suppliers must provide products and technical service to not only provide superior value in the primary use, but also to reduce the amount of chemicals the industry requires, providing practical and economical methods to reuse the chemicals and providing technology to dispose of residual chemicals in an environmentally responsible manner.

In the case of textile warp sizing, this means that sizes should provide high weaving performance, work well in existing desizing and finishing ranges, provide low levels of size required, be recoverable and reusable, and be biodegradable in wastewater treatment systems.

At Du Pont, we have long believed the best way to satisfy these requirements is to recover and reuse sizing chemicals. This offers the advantages of superior weaving performance, eliminates most of the chemicals from waste treatment, and offers significant cost savings. We not only offer ELVANOL* polyvinyl alcohol (PVA) and sizing technology, but also PVA and caustic recovery equipment through our Du Pont Separation Systems group.

This paper will discuss the general technology required to recover and reuse warp sizing chemicals. Future articles will be issued on other topics related to chemical recovery and reuse in textiles.

The economic and environmental justifications for warp sizing recovery have been discussed previously, including Warp Size Recovery by Jack Combs, 1985 Auburn University Short Course Proceedings; Carl Hoffman, 1981 American Association of Textile Colorists and Chemists Warp Size Symposium; "Technological and Economic Aspects of Recycling Sizing Agents" by Dr. J. Trauter, Meilliand Textileberichte, January, 1990.

HISTORY

Warp size recovery has been practiced in the United States for roughly 20 years. To date, only PVA has been recovered in large quantities.

Initial systems were installed by Koch Membrane Systems, Inc., (ABCOR) and Gaston County Dyeing Machine Company. The first systems were mainly aimed
at sheeting mills. These systems were installed primarily as a means to reduce biological oxygen demand (BOD) and chemical oxygen demand (COD) loading to waste treatment systems. This was an economically attractive method to respond to more restrictive waste treatment regulations. While the associated reduction in sizing chemicals cost was secondary, the savings have been very significant.

In the past five years, there has been a worldwide resurgence in interest in size recovery due to increasing environmental regulations, coupled with rising size costs.

New recovery systems have recently been installed in the United States, Germany, Taiwan, and Egypt. These newest systems have been supplied by Du Pont Separation Systems from the United States and GTV from Germany.

GENERAL CONFIGURATION

All of the systems mentioned utilize ultrafiltration as the means of separating the PVA from desizing liquor. A few of these systems recover PVA/CMC blends.

In most cases, the ultrafiltration (UF) system is installed in a finishing mill (Figure 1). The desize liquor is taken from the washers and sent to the UF system.

This desize liquor contains not only PVA, but also oils and waxes from the cotton, spin finishes from any synthetic components, and any other contaminants that have been introduced in the yarn or fabric. Solids concentration varies, but is most commonly 1.5 to 2.0%.

The weak feed is filtered to remove lint and other particles, then is introduced to the UF stages. The number of stages required depends on many factors, including the feed stream solids concentration, the desired concentration in the reclaim delivered back to the sizing area, and volume of desizing liquor to be processed.

The concentrated liquor (often called reclaim, concentrate, or regenerate) is sent back to the sizing area. If the finishing mill is remotely located from the weaving mills, the reclaim is transported in insulated trucks and pumped into insulated, heated storage tanks for reuse at the sizing area.

This reclaim still contains a portion of contaminants, primarily natural oils and waxes from the cotton. It is normal to find that 80-90% of the solids in the reclaim is PVA, and the rest contaminants. The total solids concentration depends on system design and operation, and varies from around 8% to approximately 12%. This desired final concentration must be selected prior to the UF system design and is determined primarily by sizing area needs.

I will show how the reclaim is used later.
ULTRAFILTRATION TECHNOLOGY

With the exception of one specialized chemical coagulation process, all of the suppliers of size recovery equipment use ultrafiltration. The ABCOR systems use spiral wound membranes, Gaston County uses carbon tubes, GTV has polymeric sleeves inside a stainless steel support. The Du Pont Separation System (DSS) utilizes an inorganic membrane on stainless steel support. A simple schematic of the technology is shown in Figure 2.

The porous stainless steel tube is made from stainless steel powder, which is formed under heat and pressure, then heated in an inert atmosphere to weld the powder fragments into a rigid, porous tube. These tubes are various sizes for different industries. In the case of PVA recovery, these tubes are 5/8-inch diameter.

These tubes are placed in a larger stainless steel housing, in an arrangement much like a tube sheet heat exchanger. Figure 3 shows one open DSS module. This is a 24-inch diameter, 20-foot long module from a corn processing unit. The technology is the same as for PVA recovery, only larger. The inorganic membrane is formed inside the porous stainless steel tube.

In operation, the weak feed from the desize range is fed into the porous tubes under pressure. The low molecular weight portion passes through the membrane and porous tube. The permeate is collected in the outer housing and is either reused in the washing step or disposed of.

The large molecular weight fraction is rejected by the membrane and stays inside the tube. As the liquor flows through the tube, it becomes more and more concentrated. The final concentration is determined by how long the concentrate is recycled through the membranes.

Most systems use a refractometer to control the final solids concentration. The Du Pont system uses an in-line viscometer for improved reliability and consistency.

There are several different membranes in use in the Du Pont system, depending on the application (Figure 4). These systems are used in several industries besides textiles. Within textiles, for caustic recovery and indigo dye recovery, the microfiltration membrane is used; ELVANOL* PVA recovery uses the ultrafiltration; and dye elimination/recovery has been demonstrated with the hyperfiltration membrane.

In all of the Du Pont systems, the membrane can be easily replaced in case of fouling or excessive wear. A clean-in-place/form-in-place equipment is provided to chemically strip the membrane and any fouling chemicals, then to reform the membrane in the entire system -- all without disassembly.

In actual mill practice, a routine water flush is all that is required on an on-going basis. The membranes need to be reformed only on a one- to two-year frequency.
UTILIZING RECLAIMED ELVANOL® PVA FORMULAS

There are a few fundamental items to keep in mind when using recovered PVA solutions.

The first is the reclaim must be kept hot to avoid biological activity, or inhibited with biocide. Agitation is suggested to avoid stratification in the storage tanks.

The reclaim is not pure PVA. This must be taken into account by raising solids concentration in the size box, as compared to virgin PVA, which will result in higher add-ons. Typically, in sheeting, add-ons are 1.0 to 1.5% higher with reclaim than with virgin PVA.

There will always be a need to add virgin PVA as a make-up stream to replace losses. Current recovery efficiencies vary, depending on system design and operation. This is a measure of the amount of PVA that is returned to slashing versus the total amount used, and includes losses in the sizing area, the weave room, desizing, and ultrafiltration. A well-maintained and operated system in a finishing mill should provide approximately 80% recovery efficiency.

So, one can see that 20% or more of the PVA used in slashing will need to be virgin powder.

This can be done in one of two ways.

First, if there are several remote greige mills, the ones farthest from the UF system can use all virgin PVA. The mills closer to the UF system would then use pure reclaim, with no virgin PVA added. If this method is used, the solids concentration of the reclaim must be at least as high as the highest required size box solids in slashing. There is no way for the size area to increase the solids concentration.

The second method to add the virgin PVA make-up stream is to add some to each cook made. In this method, the proper cooking procedures must be followed.

First, a small amount of cold water is added to the cooker. Recirculating pump and agitator is started. Feed in the dry PVA and form a slurry. Pump the hot reclaim into this slurry, add the proper amount of wax, and then raise the temperature to 190°F. Hold for 15 to 20 minutes.

This method can be used to either raise or lower the reclaim solids to the required size box solids. To raise the solids, use a small amount of cold water. To lower the solids, use a larger amount of water.

The amount of kettle wax added can be significantly reduced. Because of the natural waxes and oils that are in the reclaim, it is fairly typical to use half the amount of wax used in the virgin PVA formulas.
One example of formulas for virgin size and recovered size follows:

<table>
<thead>
<tr>
<th></th>
<th>Virgin Size</th>
<th>Recovered Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cold Water, Gallons</td>
<td>260</td>
<td>70</td>
</tr>
<tr>
<td>Dry ELVANOL*, Pounds</td>
<td>200</td>
<td>50</td>
</tr>
<tr>
<td>Flake Wax, Pounds</td>
<td>14</td>
<td>3</td>
</tr>
<tr>
<td>Reclaim, Gallons*</td>
<td>210</td>
<td></td>
</tr>
<tr>
<td>Add-on, Percent</td>
<td>11</td>
<td>12</td>
</tr>
</tbody>
</table>

*Reclaim is 10% total solids, 8.5% PVA concentration.

These formulas provide essentially identical weave room performance. In some cases, the reclaim formula slashes and weaves better, largely due to easier running at the slasher.

RECENT DEVELOPMENTS AND FUTURE PLANS

This has been a basic discussion of how PVA recovery systems work and how to reuse the reclaim. Future articles will outline several recent developments and continuing progress in ultrafiltration technology and sizing technology to provide easier or more efficient operations.

This will include:

- A greige mill installation so companies without integrated finishing mills can recover PVA.

- Demonstration of PVA recovery in a towel mill.

- Size blends and additives to provide easier slashing when necessary.

- Caustic recovery.

- Dye elimination/recovery.

*Du Pont Registered Trademark
FINISHING MILL WITH RECOVERY

WOVEN GREIGE GOODS → DESIZING - WATER ONLY → SCOUR AND WASH → MERCERIZE → BLEACH

UF SYSTEM → STORAGE → TRUCK

PRINTING

DYE AND WASH

FINISHING AND DRYING → WWT → FINISHED FABRIC

Figure 1
How the technology works: The membrane device separates a liquid stream into two components; a stream concentrated in suspended or high-molecular weight dissolved solids and a clean permeate stream.

Du Pont Separation Technology
Membrane Applications in the Textile Industry

![Graph showing removal ratings and relative capacity]

- **Removal Ratings (microns):**
  - $>10^0$
  - $>10^{-1}$
  - $>10^{-2}$
  - $>10^{-3}$
  - $>10^{-4}$

- **Relative Capacity:**
  - 1
  - 2
  - 3
  - 4
  - 5
  - 6
  - 7
  - 8
  - 9
  - 10
  - 11

**Applications:**
- Porous Metal, PM
- Ultrafiltration, UF
- Micro Porous, UF
- Hyperfiltration, HF
- PVA Recovery
- Dye Removal
- Casella Recovery

**Figure 4**