

When Less Is More

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Synthetic fibers facility uses thermal evaporation technology to recover waste solvent and minimize residues

Increased production of industrial sludge and more stringent disposal restrictions have created a tremendous incentive to develop cost-effective technologies that minimize residues, many of which are highly viscous and difficult to handle.

One technology receiving increased attention is evaporation, a separation technique that relies on heat to vaporize organics, which typically are recovered via condensation, and reduce the volume of material requiring disposal.

Heat can be applied directly or indirectly. Direct heating

systems, in which the heat-transfer medium — usually hot gas — comes in contact with the waste, typically are used to treat nonhazardous materials. In indirect systems, the heat-transfer medium — usually hot water, steam or oil — does not contact the waste being treated. These systems usually are used for toxic or hazardous materials and when air emissions are a concern.

Solvent recovery from waste polymer solutions. Indirect thermal processing turned out to be the technology of choice for a synthetic fibers manufacturing facility whose off-spec polymer solution was causing serious waste-handling problems.

The waste material is highly viscous and contains approximately 65 percent dimethylacetamide (DMAC). The off-spec solution is unusable and costly to dispose as a hazardous waste. It tends to harden when exposed to air, making atmospheric processing impractical. Previous attempts to recover the solvent using a combination solvent extraction and gravity separation technique were inefficient and labor-intensive.

FIGURE 1
Specific Evaporation Rate as a Function of Temperature

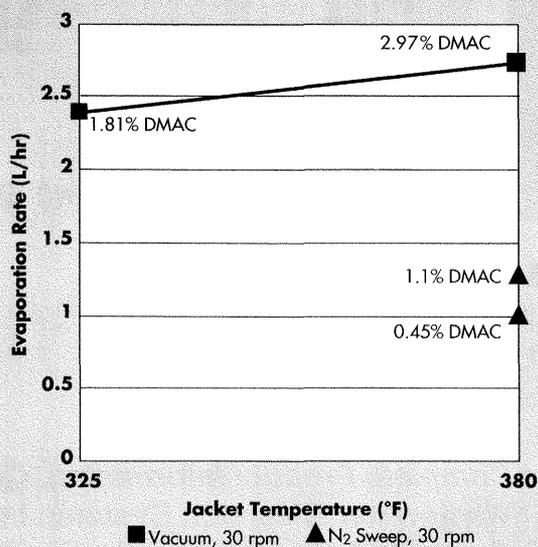


FIGURE 2
Specific Evaporation Rate as a Function of Shaft Speed

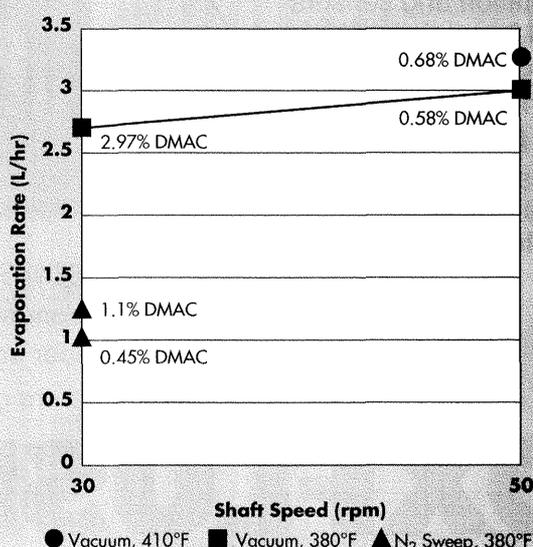


TABLE 1
Evaporation Technologies

| Technology | Advantages | Limitations | Cost* |
|-------------------------------|--|---|-------------------|
| Belt Dryer | <ul style="list-style-type: none"> • Long retention time • Handles heat-sensitive products | <ul style="list-style-type: none"> • Direct contact between heating medium and product • Large volume of vapor discharge results in high cost for solvent recovery or pollution abatement • Requires large space • Only operates at near-atmospheric pressure | 3 |
| Extruder | <ul style="list-style-type: none"> • Very high power input • Intensive mixing • Usually operates under positive pressure | <ul style="list-style-type: none"> • Limited vapor disengaging space • Power cost vs. cost of other heating media • May degrade heat-sensitive products • Requires large space | 1 Highest Cost |
| LIST Thermal Processor | <ul style="list-style-type: none"> • Large, self-cleaning heat transfer surface • Operates under pressure or vacuum • Good mixing, kneading • Long retention time • Large vapor disengaging space | <ul style="list-style-type: none"> • Improper shutdown procedures can overload drive • Foreign objects can cause mechanical damage | 2 |
| Thin-film Evaporation | <ul style="list-style-type: none"> • High heat transfer coefficient • Good vapor-handling capacity • Operates under pressure or vacuum | <ul style="list-style-type: none"> • Cannot produce dry solids • Solids adhere to high-speed rotating parts, causing unbalance • High cost of maintaining high-speed rotating equipment | 4 Lowest Cost |

* Cost rankings relative to competing technologies, with 1 being the most expensive and 4 the least expensive.

The goal, therefore, was to identify a process that would recover DMAC from the waste polymer solution and leave behind no more than 1 percent residual solids — the legal limit for disposing the material in a sanitary landfill.

Given these parameters, the company decided to evaluate four evaporation technologies: a belt dryer, a thin-film evaporator, a twin-screw extruder and the LIST thermal processor. (Table 1 summarizes the strengths and weaknesses of the technologies evaluated.) The LIST processor eventually was selected for a series of pilot-scale tests.

The polymer solution had a solids content between 25 percent and 35 percent and a viscosity of 500,000 centipoise.

DMAC's boiling point is 331 degrees Fahrenheit under atmospheric conditions; its specific heat is 0.48 calories per gram degrees Celsius; and its heat of vaporization is 119 calories per gram.

The processor used in the tests was a horizontally agitated vessel known as the Discotherm B, or DTB, developed by Swiss engineers at LIST AG (Arisdorf, Switzerland). The technology, supplied by LIST Inc. (Acton, Mass.), is patented in the United States and Europe.

The cylindrical body has a single shaft on which are mounted disc segment blades with peripheral angled mixing and transport bars. The outer casing, shaft and blades are

TABLE 2**Trial Results Using the LIST Thermal Processor**

| Trial No. | Initial Solids Content (%) | Jacket Temperature (°F) | Reactor Pressure | Mixing Speed (rpm) | Final DMAC Content (%) |
|------------------|-----------------------------------|--------------------------------|-------------------------|---------------------------|-------------------------------|
| 1 | 20-25 | 380 | N ₂ Sweep | 30 | 0.45 |
| 2 | 20-25 | 380 | N ₂ Sweep | 30 | 1.09 |
| 3 | 20-25 | 325 | 24 mm Hg | 30 | 1.81 |
| 4 | 35 | 325 | 24 mm Hg | 50 | 8.64 |
| 5 | 35 | 380 | 24 mm Hg | 50 | 0.58 |
| 6 | 35 | 380 | 24 mm Hg | 30 | 2.97 |
| 7 | 35 | 410 | 24 mm Hg | 50 | 0.68 |

heated indirectly using steam, hot water or a heat-transfer fluid. The bars continuously sweep the shell surface to provide mixing in the processor. In continuous units they also transport material through the system in an effective, controlled plug flow.

Counter hooks mounted on the body shell scrape the shaft and blades. Disc segments and counter paddles interact to produce a gentle, yet intensive, mixing and kneading action that renews the product surface for optimum heat and vapor trans-

fer and prevents product caking on heating surfaces. The ratio of heating surface to product volume is high — 10 square meters per cubic meter to 40 square meters per cubic meter. Operating under vacuum allows the process to run at less-than-atmospheric temperatures, which effectively lowers the solvent's boiling point and reduces the heat requirements.

Experimental conditions. The DTB was heated by hot oil (Therminol 66) at temperatures between 325 degrees and 410 degrees Fahrenheit. Condensate was recovered with a

tap water-cooled condenser. The unit was powered with a hydraulic drive (5 kilowatts), and the agitator was adjustable between zero and 70 revolutions per minute (rpm). Power uptake was indicated by a "J"-type thermocouple on one of the hooks. Off-spec polymer was released by gravity flow into the processor, whose fill level was estimated visually.

Seven trials were conducted, during which such conditions as mixing speed, jacket temperature, initial solids loading and fill level were varied to generate data for a full-scale unit. The system was operated at atmospheric pressure and under vacuum. The amount of solvent recovered, bed temperature and power uptake were measured as a function of batch time. The final DMAC content of the solids was measured by gas chromatograph.

During the tests, jacket temperature varied from 325 degrees to 410 degrees Fahrenheit, and agitator speed was set at either 30 rpm or 50 rpm. The reactor was run under a nitrogen sweep during the first two trials and under vacuum for the others.

Trial results. All trials produced dry, free-flowing solids with a DMAC content of 0.45 percent to 8.64 percent, indicating that this particular unit could achieve the target of less than 1 percent DMAC in the solids (Table 2).

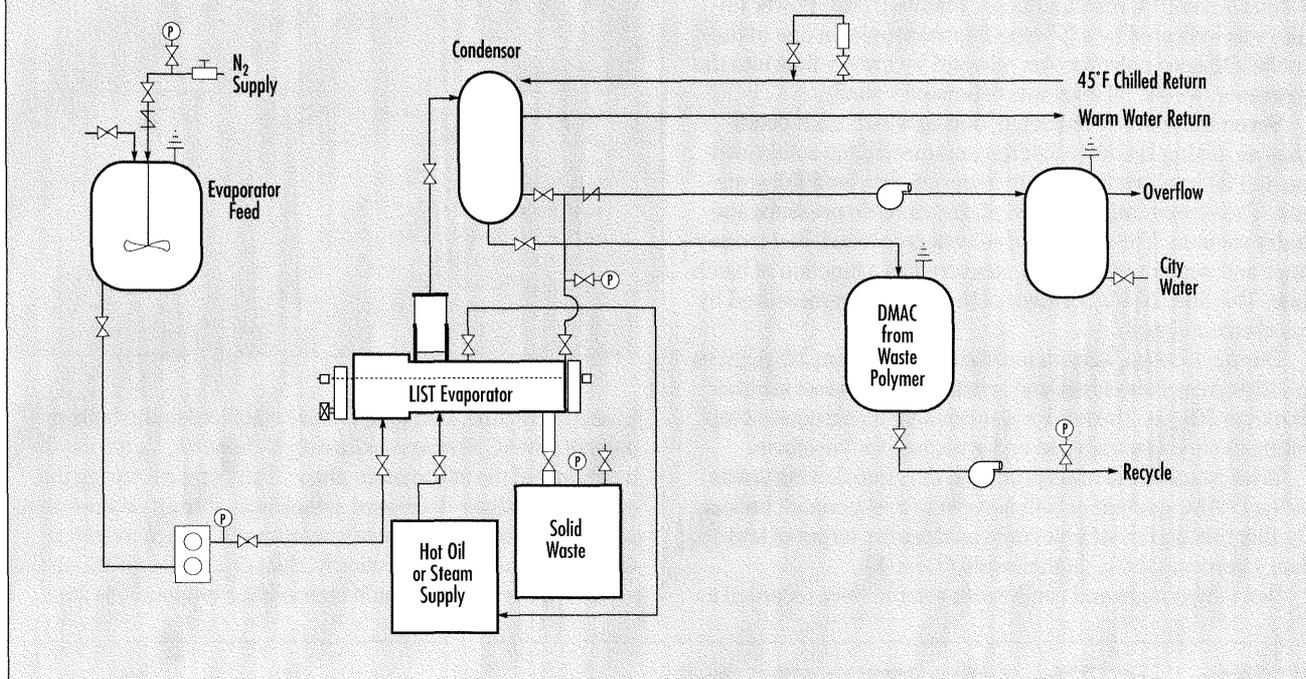
Bed temperature and condensate volume were recorded as

TABLE 3
Process Scale-up

| Description | Value |
|-------------------------------------|--------------|
| Hot Oil Heating Temperature (°F) | 380 |
| Steam Heating Pressure (psig) | 175 |
| Feed Solids (%) | 35 |
| Feed Rate (lb/hr) | 85 |
| Guaranteed Feed Rate (lb/hr) | 54 |
| Solvent Evaporation Rate (lb/hr) | 55 |
| Residence Time (hr) | 1 |
| Discharge Rate (lb/hr) | 15 |
| Solvent Concentration in Solids (%) | <1 |

a function of time. Bed temperature stabilized at the boiling point of DMAC after approximately 50 minutes, signifying the beginning of the evaporation phase. Temperature during the evaporation phase decreased significantly during vacuum operation, which increased the temperature differential between the bed and jacket. Theoretically, this means that operating the system under vacuum would increase the evaporation rate.

FIGURE 3
Full-scale Thermal Evaporation System



Hydraulic pressure was measured to provide an indication of power uptake on the agitator. Hydraulic pressure varied between zero and 30 pounds per square inch during the trials and typically peaked during the transition from viscous material to dry solid (pasty phase).

Trials 3 and 4 were run at 325 degrees Fahrenheit to simulate the temperature that would result if 80 pounds of steam were supplied as the heat-transfer medium. This temperature proved insufficient to reduce the DMAC content of the solids to less than 1 percent.

The purpose of trials 5, 6 and 7 was to determine the influence of jacket temperature and agitator speed on the DMAC content of the final product. Trial 5 was run at a jacket temperature of 380 degrees Fahrenheit and an agitator speed of 50 rpm. Trial 6 was carried out at the same temperature and an agitator speed of 30 rpm. Hydraulic pressure was higher at 50 rpm, and solids analysis revealed that the DMAC concentration was lower for Trial 5 (0.58 percent) than for Trial 6 (2.97 percent).

During Trial 7, jacket temperature was raised to 410 degrees Fahrenheit, and the agitator speed was set at 50 rpm. These conditions did not reduce the final DMAC content of the solids (0.68 percent), but a lower peak hydraulic pressure was recorded during this trial, probably because the material was less viscous.

Specific evaporation rates were estimated for each trial based on the volume of solvent recovered, the batch time and a heat transfer area of 0.31 square meters in the processor. Specific evaporation rates ranged from 1 liter per hour to 3 liters per hour.

Temperature had a dramatic effect on evaporation rate,

which increased at higher temperatures and nearly doubled when the system was run under vacuum (Figure 1).

The evaporation rate also increased as the agitator speed increased (Figure 2), but temperature had a more dramatic effect, indicating that the process is heat-transfer limited.

Full-scale plant. The measured evaporation rates from Trial 5 were used to scale up the system to the desired feed rate of 30 pounds per hour. Estimates indicated that a 40-liter DTB has more than enough capacity to handle this feed rate (Table 3).

A flow diagram for the full-scale system is shown in Figure 3. Waste is fed to a continuously stirred heating tank blanketed with nitrogen before being pumped to the jacketed evaporator, which is heated with steam to approximately 380 degrees Fahrenheit. Free-flowing solids are discharged continuously under vacuum to a waste hopper, and vaporized solvent is condensed and recovered. The unit features intensive mixing and surface cleaning, which help maximize heat transfer.

The full-scale system, which has been on-line since October 1993, consistently produces a residue whose DMAC content is less than 1 percent.

Based on solvent recovery and a reduction in disposal costs associated with rendering the solid residue nonhazardous, pay back on capital investment is estimated to be 2.5 years. The unit has dramatically reduced the amount of operator attention and downtime associated with the solvent recovery process. ■

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