Chapter 5/Part 2: Practical Application of Reactive Dyes

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Abstract

The various methods by which reactive dyes may be applied batchwise, continuously or by the hybrid pad batch method are discussed. The chemistry of reactive dyes is discussed in terms of both the reactive groups and the chromophores to which they are attached. The means by which dye manufacturing companies might reduce fixation inefficiencies and effluent problems are covered by means of examples which show the amount of work involved. The benefits of bifunctional dyes are examined.

Key Terms

Batch Dyeing
Bifunctional Dyes
Chromophores
Continuous Dyeing
Fixation Efficiency
Heterocyclic Rings
Leaving Groups
Nucleophilic Reactions
Pad Batch Dyeing
Physical Form
Reactive Dyes
Reproducibility
Vinyl Sulfones

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Experimental

To restate what has been said before (1), fiber reactive dyes practically classify themselves by their reactivity (reactive groups) and the optimized conditions in which they are best used. There is no simple alternative to taking at face value most of the technical information from the suppliers. If, on the other hand, good dye application laboratories are available, it might be possible to discover that some dyes from different groups perform satisfactorily together under particular conditions of use.

Padding applications are especially sensitive to the selection of dyes which do not have the same strike rate and tailing characteristics. For a general treatment, see the section on padding in Chapter 4, which deals with sulfur dyes. However, there are situations here which do not apply to sulfur dyes. Sulfur dyes have a limited number of colors to choose from, and each one must be fully utilized.

It is both practical and important to choose mixtures of reactive dyes which are mutually compatible (behave in the same way) from among the wide variety of products available. This compatibility is more important than in the case of sulfur dyes, where different formulas can be calculated for both the pad bath and feed tank to compensate for dyes that behave differently. For reactive dyes, it would be desirable to be able to calculate the amount by which to underfeed or overfeed the pad. The author is not aware of any fundamental work in this area, although there are some qualitative methods which involve observing the depth and hue of single and multiple paddings to determine the compatibility of reactive dye mixtures.

Batch Dyeing

The mechanism of reactive batch dyeing has been discussed at some length, but no account has yet been taken of the variety of procedural variations which may be practiced in particular circumstances. These are the result of differences in dye selection, dyeing equipment and controls, and to some extent, the preference of the dyer.

However, before the dyeing can commence, the question of preparation rears its head, as it always does.

Preparation

At the expense of sounding like a broken record, it should be said, over and over again, that quality preparation is a prerequisite for quality dyeings (1). Anything less introduces an element of risk which, while it may be acceptable for some goods some of the time, will not be acceptable for all goods all the time. This is particularly important for woven piece goods to be dyed with the most reactive (cold dyeing) dyes, and less so for some open knitgoods, which it is sometimes possible to dye with less reactive dyes (hot) in the greige state. At least knitgoods will not contain starch or other sizing materials containing hydroxy groups which may also react with the dyes (2).

Preparation can include any of the following processes: scouring, desizing, bleaching, causticizing and mercerizing. Any of the last three could leave residual alkali in the goods, which must be removed completely if accurate control of the alkalinity and therefore of the reactive dyeing, is to be achieved. For some goods, provided the dyes are stable to the treatment, post-bleaching with peroxide or other per-compounds might be acceptable.

Many of the chemical auxiliaries used in preparation should be screened before use, for they can have adverse effects on the dyeing process if not completely removed. These can include: organic chelating or sequestering agents which may remove the complexing metal from pre-metallized reactive dyes; surfactants, which may be incompatible with the dyes and can retard dyeing or produce dye spots; and residual oxidizing agents from the bleaching.

On the subject of oxidants, it is not generally recognized that the amount of chlorine in municipal water supplies has sufficient oxidizing character, if more than about 0.5 parts per million are present, to destroy significant amounts of dye and affect pale shades. Heating the water, or adding 0.1 grams per liter sodium thiosulfate, effectively eliminates the problem, but only if done before the addition of the dye.

Quality control testing for the residual pH of the fabric and the amount of water and solvent extractables can be helpful (3), but samples with low extractables are not absolute assurance of good preparation if the residuals are unevenly distributed throughout the goods.

Dyeing Procedures

These are often split into four different categories:

- Traditional or conventional: This is the method, discussed at length earlier, in which the temperature is raised to fixation temperature and the dye exhausted with salt, prior to the addition of alkali. It should be apparent that, if the rate of salt addition and temperature rise are not controlled carefully, uneasiness may result.

- One Step or All-In: Here all the ingredients are circulated together cool (alkali included) prior to the heating of the bath to the dyeing temperature. The method, when compared with the conventional method, has additional drawbacks. Maintaining the starting temperature is critical for repeatability; controlling the
Reactive Dyes

rate of heating is essential for levelness, since both exhaustion and irreversible fixation are occurring simultaneously from the start, and there is a possibility of lower color yield. Why do it? It is the simplest and quickest method available, since no additions need to be made during the process.

- **Constant Temperature:** In this method, dyeing is carried out at the fixation temperature, thus completely eliminating any problems due to improper control over the rate of rise of temperature. Salt may be added at the start, or in portions, and the alkali is added after a suitable time interval. The procedure is about as controllable as you can get.

- **High Temperature:** To ensure better leveling and migration, which is always improved by elevating the temperature (Chapter 1), it is possible to commence dyeing at a higher temperature than that of fixation, with the salt in, and allow the temperature to fall to the fixation temperature before the addition of alkali. The principle use of this method is for fabrics from high twist yarns, tightly woven goods, or viscose rayon fabrics.

- **Washing Off:** Covered earlier in Part 1, this critical process is one which can make or mar the overall shade and fastness of reactive dyeings, see (4, 5).

**Continuous Dyeing**

Here again, there are four principle application methods, each with its own unique aspects. They are pad-steam, wet-on-wet (pad-paddle), dry-pad-paddle, and pad-dry-cure. Of these, the conventional method for 100% cotton is the pad-dry-cure process, this has found its greatest use in the Terry cloth area. The process is not efficient, but on the other hand, neither is the intermediate drying of goods which can have 100% wet pickup.

**Pad-Dry-Cure:** This process is an interesting curiosity. Some reactive dyes may be padded onto cellulose in the presence of sodium bicarbonate and urea. Under the severe conditions of thermal fixation and the presence of water, the bicarbonate generates carbonate. While it is normal to expect water in a thermal oven, it is sufficiently retained due to the extremely hydroscopic (water attracting) nature of the urea. There is sufficient water to permit both alkali formation and dye-fiber reaction. However, some undesirable smoke generation and yellowing of the cellulose can occur. This is definitely a special case procedure. Urea is also used as a pad bath additive for increasing the solubility of sparingly soluble dyes.

Note: Whenever cellulose is heated in the presence of alkali, there is always the possibility of reduction, cf. glucose/alkali reducing systems for sulfur dyes. However, if there is sufficient reduction potential to cause loss of color with fiber reactive dyes, the addition of the mild oxidant, sodium metanitrobenzene sulfonate, should alleviate the problem.

With reactive dyes, there is also the economically interesting possibility of dyeing by a semi-continuous process known as the pad batch process.

**Pad Batch Dyeing**

This important process represents the minimum possible investment for dyeing large yardages of cellulose fabrics with cold dyeing, highly reactive dyes. The goods may be woven or knit, tubular or flat.

The equipment required is a suitable paddle and for many fabrics a series of A-frames supplied with perforated rolls which can be mechanically rotated, and to which a water supply can be attached. However, the perforated beams (rolls) are often replaced by boxes into which the goods are batched, particularly in the case of tubular knit goods.

In principle, all that is required by the process is to pad on dye and alkali (a wet pickup of about 135% is desirable) and to batch the goods as uniformly as possible. The goods are then wrapped with plastic, to prevent evaporation and the reaction of caustic alkali with the carbon dioxide in the air, beams are rotated to prevent preferential drainage, and the goods are stored in a temperature controlled area to help ensure lot to lot reproducibility.

Under these conditions, provided the fabrics are not bathed too nonuniformly, the dye will react slowly but evenly throughout for whatever optimum time is dictated by the alkali and reactive dye selected (2 to 48 hours) (4). The goods can then be efficiently washed off on the perforated beams with a slow flow of hot or cold water. However, many prefer washing off in conventional batch or continuous dyeing equipment. Certainly it is easier to make adds to a large volume of liquor.

It has been found that pad batch dyeing greige goods with or without post-bleaching can improve hand and appearance while reducing costs and process times substantially.

The pad batch process offers particular benefits to those dyeing polyester/cotton fabrics who wish to use their expensive, high temperature, high pressure jet dyeing machines for dyeing the polyester portion without carriers, and not for lengthy reactive dyeing processes for the cotton, which do not require temperatures even as high as the atmospheric boil.
Chemical Stripping

Once reactive dyes have been applied and leveling or stripping by simple physical (highly reactive), there is no longer a possibility of removing, there is no longer a possibility of what steps must be taken to deal with the problem.

For heavy shades, a reduction of about 20-40% in depth of shade can sometimes be achieved by hydrolysis of the dye-fiber bond with alkali in the presence of salt at 95°C (200°F).

Depending on the individual dyes, complete chemical stripping (color destruction followed by oxidation; oxidation followed by reduction. An example is given below (4).

If premetallized reactive dyes are present, it is first desirable to remove any complexing metal by treatment of the dyeings at the boil with ca. 2 grams per liter EDTA solution followed, if necessary, by a boil in 5 grams per liter each of caustic soda and sodium hydrosulfite. Twice as much soda ash may be substituted for the caustic soda. A cold oxidative treatment with sodium hypochlorite should follow the reductive stripping, and all traces of chemicals should be removed prior to any redyeing attempts on a sample of the stripped goods.

Chemistry of Reactive Dyes

Reactive dyes, apart from easily recognizable reactive functional groups, have structures which are not very different from those of acid dyes and direct dyes in several important respects. Direct dye molecules tend to be larger, more linear and somewhat less brightly colored than acid dye molecules with specific components such as J-acid derivatives being much in evidence. Otherwise, it might be tricky to tell acid and direct dye molecules apart.

Chromophores

Reactive dyes all rely on many of the same chromophoric groups as acid and direct dyes for their color, although the azo chromophore, \(-N=N-\), is by far the most important, cf. Fig 1a, 3, 4 and 6.

Any of the following chromophores may be used: mono and disazo (metallized and unmetallized), triphenoxazine and phospholycyanine (see Chapter 2). Acid reactive dyes, especially bright blues, often depend on anthraquinone derivatives (see Chapter 3 on Vat Dyes and Fig. 1b). All reactive dyes contain sodium sulfonate groups for solubility, and dissolve in water to give colored sulfonate anions and sodium cations. Most reactive dyes have from one to four of these sulfonate groups.

Reactive dye molecules, however, do have several specific structural (reactive) features of their own. These are of two principal types: carbon-nitrogen (heterocyclic) ring structures and masked vinyl sulfones.

Carbon-Nitrogen Rings

The reactive structures (R), of which some important examples are shown in Fig. 1, are attached to the rest of the dye molecule by bridging groups (B) such as imino, \(-\text{NH}-)\text{ substituted imino and amide linkages, }-\text{NHCO}-\text{. The leaving groups (X) are attached initially at carbon atoms adjacent to a ring nitrogen. The carbon atoms are positively polarized and come under attack from nucleophiles such as the celluloseate and hydroxide anions. The outcome is the nucleophiles substitute themselves in place of the leaving groups, which are expelled carrying an electron with them, usually as inert anions. Potential leaving groups (X) include chloro groups, fluoro groups, \(-\text{F},\text{ and methyl sulfonyl groups, }\text{CH}_3\text{SO}_2-\text{, arranged in order of decreasing importance. These leaving groups may be called }\text{nucleofugic because they take refuge by running away from nucleophiles. This might be an appropriate place to list the names and abbreviations for a number of these reactive species, arranged in order of gradually increasing reactivity: trichloropyrimidinyl, TCP; monochloro-triazine, MCT; masked vinylsulfones, VS; monofluorotriazine, MFT; difluorochloropyrimidine; FCP; and dichlorotriazine, DCT. All the heterocyclic compounds are shown in Fig. 2. Vinyl Sulfones

These reactive groups are normally transients, and are masked by water solubilizing leaving groups and their name is abbreviated to (VS). Vinyl sulfone groups are actually written: \(-\text{SO}_2-\text{CH}==\text{CH}_2\text{, but this group is rarely if ever found in a reactive dye product. What is found is generally the functional group: }\text{SO}_2-\text{CH}_2-\text{CH}_2-\text{SO}_2-\text{(H+ or Na+).}

Whether the structure includes the hydrogen ion or the sodium ion depends of the pH. The bridging group (B) is the sulfone, \(-\text{SO}_2-,\text{ portion of the structure, which withdraws electrons from the rest of the group so that, in the presence of alkali, the leaving group (X) departs with a hydrogen ion (from the carbon next to the sulfone group) and unmask the hidden vinyl group: }\text{SO}_2-\text{CH}_2-\text{CH}_2-\text{SO}_2-\rightarrow \text{SO}_2-\text{CH}==\text{CH}_2+\text{H}^+\text{. Eq. 1

The bisulfate ion is more or less neutralized to a sulfite ion, depending on pH. The vinyl group immediately begins to add back whatever nucleophiles are around e.g., \text{OH}^{-}\text{ and Cell-O-}\text{—and finally adds back the hydrogen ion as well. This addition reaction is even more complex than indicated (6). But, although the overall reaction with a nucleophile is often referred to as a vinyl addition reaction, and although it is a multistage reaction, the initial and final state of affairs may again be reliably summarized.}}
Reactive Dyes

by the same Eq. 3 shown in Chapter 5, Part 1.

Dyestuff Research

As stated in the review, Chapter 5, Part 1, despite the demand for reactive dyes, they do currently have several noteworthy deficiencies as a group. These include the generally low efficiency of fixation, with its resulting highly colored waste, and the large amount of salt needed for their fixation. These drawbacks do not apply to all reactive dyes, but there certainly is a need to attend to them. If the problems are to be solved, they will be solved by the responsible dye manufacturers, not by their imitators, and it will cost them time and money.

One of the promising approaches appears to be in the development of bi- and polyfunctional reactive dyes. These will be explored a little here so that their potential, along with the developmental problems, may make it clear that the advantages are not to be gained without considerable effort.

Bifunctional Reactive Dyes

A few dyes with two reactive groups have been known for about 30 years. Some are based on two monochlorotriazine groups (MCT)—e.g., C.I. Reactive Red 120 (see later)—and some on two masked vinyl sulfone groups (VS)—e.g., C.I. Reactive Black 5 (see Fig. 3). The latter is a most important and relatively economical reactive dye, but those used to dyeing sulfur dyes would find the dyeing results neither economical nor very black. Recently dyes have become available which have both economical nor very black. Recently dyes have become available which have both reactive groups (see Table I).

The answer depends on three factors: probability of fixation, relative color efficiency and dyebath behavior.

- Probability of Fixation: Your average gambler will be able to tell you immediately the odds of two men, each flipping a coin simultaneously, coming up with at least one "head" between them. For each individual the probability of a "head" is 50%. But together the probability of at least one "head" is 75%.

The same principle should hold true for reactive groups. Table I, line 1, lists several probabilities (as percentages) that a particular reactive group on a monofunctional reactive dye, in any given conditions, might react with cellulose. These are called dye fixation efficiencies. The values, 50-70%, are in the normal range; cf. Fig. 3, Part 1. If there is more than one reactive group of the same reactivity attached to the molecule, the probability that at least one of them reacts is increased (Table 1, lines 2-4). Lines 2-4 in Table I show the hypothetical dye fixation efficiencies of dyes containing two to four of the reactive groups which in monofunctional dyes have 50-70% probability of reaction (line 1).

Here we can see that if three reactive groups of fixation efficiency 55% in the monofunctional dye are all present in the same trifunctional dye, the dye fixation efficiency could be 91%. The possible benefits of increasing the number of reactive groups look very promising, but the story is not quite so simple. You cannot add more reactive groups to a dye and still have the same dye!! Each time the overall structure is changed, so are the physical and chemical properties of the product.

- Relative Color Efficiency: Whenever a reactive group is added to a dye molecule there is an increase in the size of that molecule. It is now customary to refer to the relative size of molecules as their rmm, or relative molecular mass. But each molecule has a fixed number of chromophoric groups giving it the color: e.g., one azo group.

Suppose a monofunctional reactive dye has an rmm of 700 and one azo group. If another reactive group is added, the rmm may be increased by 200, to 900, but the new bifunctional dye has still only one azo group. It now requires 900 units of the new dye to have the same color as 700 units of the original dye. The relative color efficiency of the new molecule is only 77.8% of that of the original molecule. This has nothing to do with fixation efficiency!

Such potential losses in relative color efficiency, due to adding reactive groups, must be taken into consideration before deciding whether the gain in fixation efficiency (by increasing the number of reactive groups) could be worthwhile. However, there are occasions where there may be a relative color efficiency gain (see later, Table II).

- Dyebath Behavior: The acid test of any new dye is its performance in the dyeing operation. However promising it looks with respect to fixation and relative color efficiency, if it is not compatible with other dyes under comparable conditions, it is useful only for self shades.

Between having acceptable solubility, substantivity, salt sensitivity, rate of sorption, rate of diffusion, efficiency of fixation, quite apart from shade, fastness properties and cost, and being environmentally acceptable, a new reactive dye may reach the patent literature but fall short of the market.

Too Many Options

The U.S. market for reactive dyes is certainly large, but so are the numbers of products vying with one another for a share. There are presently about 200 structures, in the form of 800 products, being sold by four times as many suppliers as there are major manufacturers (7). This abundance normally results in a small production volume for any one dye (8). But anyone bringing a new dye to the market must be able to offset development costs against profits from potential sales.

The previous section, without being exhaustive, indicated the many technical criteria any new entry must meet. The following will indicate how many possible products may have to be synthesized, from which a potential new product may be painstakingly selected.

Let us take as a starting point a structural segment common to C.I. Reactive Reds 1, 3, 120, 240 and others. It is shown in Fig. 4 and will be known as segment -C-. Segment -C- contains one azo group, two sodium sulfonate solubilizing groups and one monochlorotriazinyl (MCT) reactive group. To make the segment into a complete organic dye structure requires two additional segments A- and A'-... Since A- must be an aromatic ring, such as

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<tr>
<th>Table I. Hypothetical Dye Fixation Efficiency vs Number of Reactive Groups</th>
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<td>Number of Reactive Groups</td>
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</tr>
<tr>
<td>1</td>
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<td>2</td>
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<th>Table II. Bi- and Polyfunctional Reactive Red Possibilities</th>
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<td>Structural Type</td>
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<tr>
<td>I (A-C-A')</td>
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<td>II (A-C-B-C-A)</td>
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¹Calculated relative to C.I. Reactive Red 3, 100%; based on rmm per azo group.²See reference 9.
benzene, let us have $A'$ - an aromatic ring also.

To provide for further reactive groups and additional solubility, let us specify that both $A$ and $A'$ shall be benzene rings, which may optionally be substituted with either a sodium sulphonate group (S) or a masked vinyl sulphone group ($\text{VS}$), in place of the hydrogen attached to the carbon atom at any of the three positions labeled in Fig. 5. Position 2 is called ortho (o), 3 is called meta (m) and 4 is called para (p) relative to the attachment at position 1.

Even these limited options give the possibility of 49 different red, mono-, di- (bi-) or trifunctional reactive dyes, all of which can be represented by the general formula: $A - C - A'$ (Type I). The properties of 18 of them have been recently examined (9). These are indicated in Table II.

If two segments $A - C$ are joined by group $B$ - (which may be a benzene ring attached at both ends in the 1 and 4 positions) (Fig. 6) an additional seven symmetrical red bi- and polyfunctional dyes can be added. A further seven come with every new link (-B-) between the -C- segments. All these added dyes have the general formula:

$$A - C - B - C - A'$$

In all, there are 40 different di- or trifunctional dyes possible, with only the seven substituents shown in Fig. 5 plus one group $B$ - (Fig. 6, see Table II). If the chloro groups (MCT) reactive dyes are replaced by an alternative leaving group - e.g., fluoro (MFT) - the total number of dyes would double.

A combination of relative color efficiency and dye fixation efficiency helps to point out the most promising of the candidates. Ease of manufacture and cost will take care of the rest.

Structural Shorthand: Because reactive dye structures can be a pain to draw, it is a frequently used convention to substitute a triangle for the triazine ring. The corners represent the carbon atoms which carry the leaving groups; for example, see Fig. 7.

**Reproducibility of Dyeings**

Besides a high degree of fixation, another of the properties sought in fiber reactive dyeing is that the color of the dyeings should be insensitive to unintentional or unavoidable variations in the reaction conditions.

This property has been promoted for the dyeings of the two different reactive groups MCT and VS (9). The point is made that since these groups are of different reactivity and different optimal application conditions, they will show a more similar degree of fixation over a wider than normal range of processing conditions (between those optimal for either of the two types of reactive group) than will dyeings of two identical fiber reactive groups.

It seems probable that, if the reactivity of the two different fiber reactive groups are too far apart, the chance of maximizing fixation will be surrendered in favor of an added assurance of repeatability if procedural errors are tolerated.

The author believes that vigorous procedural control, and selected bifunctional dyes with functional groups of very similar reactivity, will produce the best all around results.

**Physical Form**

Fiber reactive dyes are normally sold as fine powders, which tend to be hygroscopic (water attractive). It is important to ensure that such powders are kept as dry and free from water contamination as possible. They also tend to be prone to dusting - which is particularly undesirable because they are very highly colored and also reactive.

Contamination of goods and equipment by colored dust is clearly undesirable in both plant and laboratory environments and can lead to damaged goods, faulty dyeing recipes and other undesirable side effects. But even more important are the potential health hazards of dust which has the capability of reacting with the lining of the lungs. Dust masks or respirators are essential equipment when using reactive dyes and other special methods and equipment have been developed for proper storage and handling of such materials. Consult a reputable dye manufacturer if in doubt.

More recently, some reactive dyes have become available as liquids carefully stabilized at an appropriate pH to cut any hydrolysis down to a bare minimum. While these products are manufactured for the convenience of customers, it must be understood that they do not have an infinite lifetime, and will withstand only a limited amount of mishandling and adulteration.

**Standardization**

It is worth noting that reactive dyes cannot be reliably standardized, or the quality of incoming shipments checked, by transmittance measurement of solutions. The hydrolyzed species has similar transmittance characteristics to those of the reactive parent compound, but will not dye. Some hydrolyzed dye may be present in any reactive dye product. Analytical techniques such as HPLC (high performance liquid chromatography) are applicable, but there is no substitute for making dyeings under controlled conditions, to compare samples with known standards.

**Review**

In dyeing reactive dyes, mills must practice both dyeing and chemistry simultaneously to reap the greatest benefits from this very diverse category of dyes. Certainly no other application category has more brightly colored products to choose from. However, care must be taken to control everything that is controllable if good results are to be consistently achieved. These include: dye, alkali, salt and other chemical weighings, liquor ratios, times, temperatures, rates of change...
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### Reactive Dyes

Of temperature, circulation of dyebath, rate of salt and alkali addition, washing conditions, preparation quality and water quality not to mention proper care in handling and storage. The rewards will be good lot to lot and piece to piece shade reproducibility, and dyeings with excellent wash and good lightfastness.

With respect to their environmental impact, reactive dyes are continuously being improved, and in the hands of some few manufacturers, many of the products in use today will be superseded by products with higher fixation, which require less salt and which will have equal or better color yield and fastness properties.

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### References


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### Philadelphia College To Host Nonwovens Workshop

Philadelphia College of Textiles and Science will conduct an on-campus workshop on the technology of nonwovens August 4-6. Designed to improve the working knowledge of management, sales, and other personnel, the course will be equally divided into lecture and hands-on lab experiments. The course will be taught by Robert Wagner, professor in the School of Textiles, and Herbert Brandt, associate professor and director of the Grundy Center for Textile Product Evaluation. Information on the course is available from the PCTS Office of Continuing Education, telephone 215-951-2901.

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