

## **FOAM INDIGO DYEING OF COTTON YARNS: NEW TECHNOLOGY FOR AN ANCIENT DYE**

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Denim is being challenged partly because of its enormous volume and partly because of its dyeing method. Indigo, the unique colorant for ubiquitous blue jeans, is highly sustainable; safe enough to be widely used as a food colorant. However, the indigo dyeing *process* is criticized for poor sustainability, primarily because sulphur reducing compounds and large amounts of problematic wastewater are required with current dyeing methods. Foam dyeing is a water-saving, environmentally friendly technology that is increasingly used around the world, primarily for fabrics. Its use for indigo dyeing of denim yarns has been hindered by the fact that indigo becomes insoluble in the presence of oxygen. The research reported here developed a foam dyeing system that eliminates the oxygen until the dyeing process is completed and the yarns are ready to be oxidized. Results to date have demonstrated that speed of the dyeing process can be multiplied, dye uptake and dye fastness improved, water and energy use greatly reduced, floor space required for dyeing dramatically reduced, and all without the use of the sulphur compounds.

Denim jeans are an iconic global apparel product with annual production numbering in the hundreds of millions. Indigo is the colorant used to dye the yarns for these. One of the most ancient dyes, it was originally extracted from the leaves of plants. Indigo is unique among all blue dyes, synthetic and natural, because of its purity of colour that does not dull with age and exposure. Almost all colorants suffer progressive damage from the elements. Indigo is different because its damaged product is water soluble and simply washes off, leaving the undamaged remainder as fresh and bright as ever.

Indigo has been exactly copied synthetically for more than a century. Synthetic indigo is safe enough to be widely used as a food colorant as FD&C Blue 2. However, the indigo dyeing process is widely criticized for poor sustainability, primarily because sulphur reducing compounds and large amounts of problematic wastewater are still required with current dyeing methods. The blue indigo pigment is not soluble in water until it undergoes a chemical change called reduction (a gain of negative electrons), which applies a negative charge to indigo. This converts the large blue particles to polar, yellow ions called leuco

indigo, which are small enough to dye, in other words, enter into the interior of cellulosic fibre. In 1993 BASF, the original inventor of synthetic indigo in 1897, introduced pre-reduced indigo which is reduced using hydrogen gas in the manufacturing process. Pre-reduced indigo eliminates the sulphur compounds used for the initial reduction of indigo; however, the maintenance of reduction during the conventional open-atmosphere dyeing process still requires a sulphur compound, namely sodium hydrosulphite or “hydro” for short. The net result is that about one-half the hydro is eliminated with pre-reduced indigo. For decades (even before pre-reduced indigo) BASF and others have unsuccessfully attempted to replace this maintenance hydro with other chemicals or electrolysis. All these alternatives have proved either too costly or too difficult to use under the conditions of conventional dyeing.

All commercial methods of indigo dyeing, whether done continuously or in a closed batch, present special challenges for efficient use in modern manufacturing. When a fabric or yarn is removed from the dyebath, the leuco indigo almost instantaneously reacts with oxygen in the air and initially turns green (the combination of partial blue and partial yellow) as it transitions to the insoluble, intensely coloured blue indigo. Current methods leave significant amounts of the dye liquor on the surfaces of textile substrates. This unavoidably oxidises and leaves behind considerable surface indigo that must be removed by washing.

This reality for indigo requires that the dyeing machinery be different from the norm. The dominant technology is the ‘continuous rope-dyeing range’, which was developed in 1921<sup>1</sup>. Figure 1 shows the entry and exit points of a single vat in this dyeing range. As soon as the yarns exit the squeeze rollers into the air, the green shade appears, the indigo is fixed in the position it attained in the yarns, and dyeing is stopped. The result is that considerable dye liquor oxidizes on the yarn surfaces. This must be washed off as waste because this unfixed dye is subject to coming off and has a duller colour than the purer blue held in the interior of the fibres. Even intensive washing by the dyer cannot remove all this unfixed indigo which will later rub off or wash off progressively in consumer use.

Figure 1 shows only the front of one dye vat of a typical indigo dyeing system. There are seldom less than six such vats, with up to twelve being common. Sizes of each one of these range from 500 to 1,500 gallons of fluid capacity. These are accompanied by a multitude of guiding rollers and multiple 5,000-to-10,000-gallon storage tanks. The footprint on a factory floor may be 100 meters long.

In addition to being a slow-moving, water-intensive technology producing large amounts of salts, the dyed ‘ropes’ – consisting of about 400 individual yarns – must be re-beamed (separated into individual yarns for weaving). This is a slow, tedious task that is very labour-intensive.

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<sup>1</sup> An alternative method is called *slasher* dyeing. This method is different only in that beams of parallel yarns are dyed instead of ropes.



Figure 1. Showing a vat of the continuous rope-dyeing range. In the picture on the left, undyed 'ropes' of yarns are submerged into the front of the yellow leuco dye vat, which has a foamy, oxidized blue surface. In the picture on the right, the ropes exit through rollers that squeeze off excess dye liquor. Upon exiting the squeeze rollers into the air, the ropes immediately turn green. About a minute of exposure to air is required for complete oxidation because of the unwanted surface liquid that remains. These dyeing ranges may have up to 16 vats and be 100 metres long, depending on how dark the shade is to be.

## Objectives and Procedures

This project is to be understood as applied development of a known technology. Foam dyeing is a water-saving, environmentally friendly technology that is increasingly used around the world. Of course, ambient air is the gas commonly used in making the foam.

This research project was undertaken to develop a foam dyeing technology suitable for use with indigo dye and with yarns. The fundamental challenge was to determine the machine design parameters and process controls necessary for the foam application of pre-reduced indigo on yarns. The experimental system must enable both precision execution of the process along with measurement of the parameters that are being varied, followed by measurement and evaluation of results.

The patent-pending design consisted of three sequential chambers to ensure an anaerobic environment for dyeing:

1. A *purge chamber* that removes oxygen from the yarns before going into an application chamber. Among other things, this contributes toward eliminating the use of sulphur reducing compounds.

2. An *application chamber* that precisely controls the delivery of foam-based dye liquor to deliver the dye liquid from the collapsed foam in a manner that enables control of dye uptake and penetration.
3. A *kinetic chamber* that modulates the dye absorption to minimize the amounts of detrimental surface indigo and wasted indigo, enhance the darkness and purity of the blue, and improve the colour fastness.

Additionally, there must be adequate control of the yarns for reliable foam indigo dyeing. Yarns do not behave as a structured fabric, which is the application for which foam dyeing is widely known to be effective with reactive and other non-oxidizing dyes. Consistent quality control requires consistent control of both the yarns and the foam.

The yarn handling system was designed to deliver 40 yarns from a small beam through the dyeing system and rewind the dyed yarns onto another beam. It provides computerized control of yarn tensions from beginning to end. It was designed to reach a maximum speed of 100 yards per minute (ypm). Added to the system is a creel with computerized control of yarn tensions, so that yarn packages can be fed directly into the yarn handling system.

Fabrication of the foam generator and the three-stage, anaerobic foam dyeing system was contracted to Gaston Systems<sup>2</sup>. It delivers precisely metered foam directly to the 40-yarn sheet as it passes over a sequence of rollers. It is coordinated by computerized feedback with the yarn handling system, so that constant yarn tension is maintained throughout. It enables real-time adjustments for foam blow ratios and wet pick-up levels. It also automatically adjusts these variables if the speed of the yarn movement is adjusted.

The system was completed by the addition of an oxidation and drying rack between the kinetic chamber and the yarn take-up beam. The yarns need time to oxidise before drying, because moisture is necessary for oxidation to occur. Fortunately, oxidation occurs quite rapidly and the design of the rack is adequate even when running at maximum speed. However, the infrared drying capacity we used is generally inadequate when the yarns are running faster than 40 yards per minute; therefore, it is being increased.

Figure 2 shows a picture of the entire yarn handling and dyeing system in the laboratory at the Fiber and Biopolymer Research Institute. At the beginning is the creel loaded with yarn packages. Barely visible is a take-off beam stand, so that yarns can be fed from either creel or beam. The S-wrap assembly begins the yarn transport system. It is followed by the foam application chambers, and then the oxidation/drying rack. It ends with the take-up beam stand. The foam generator sits behind the application chambers.

The total length of this set-up in Figure 2, including the yarn creel, is only 15 metres. Making the machinery wider would increase the yarn capacity, so that

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commercial scaling of the system would not greatly increase the total footprint of the system.

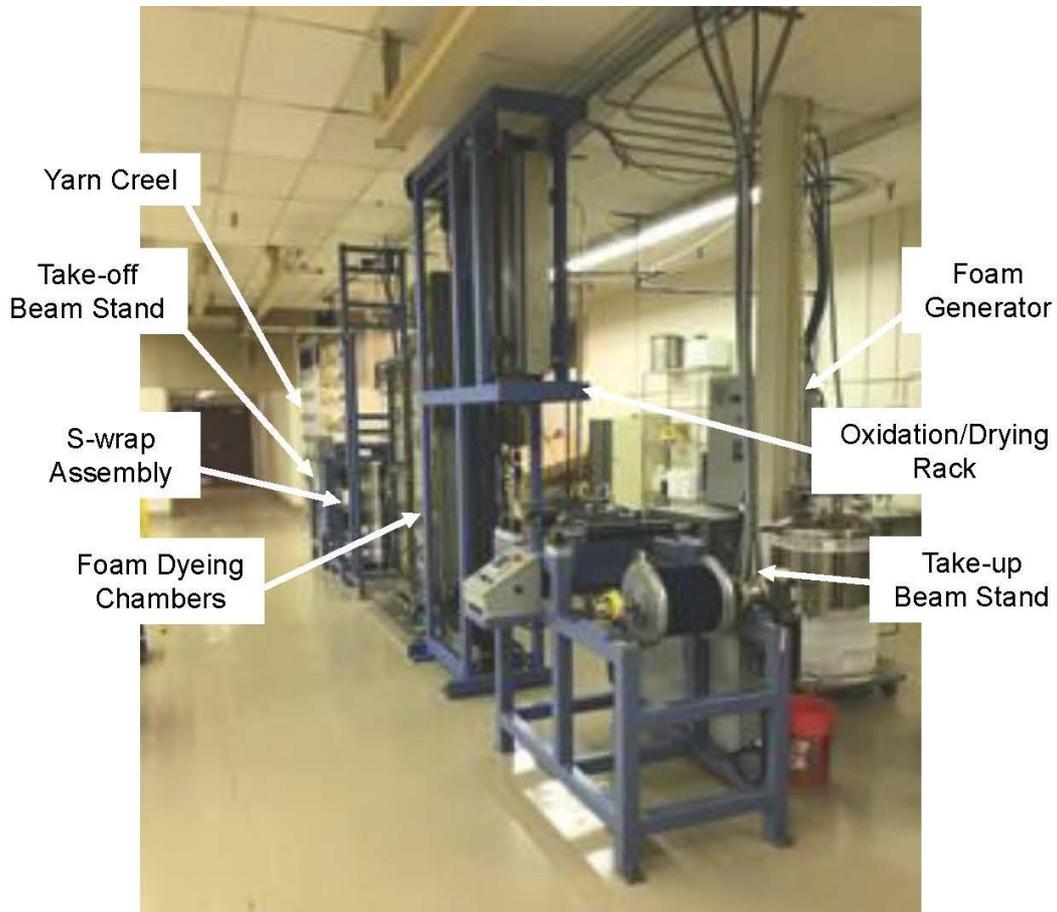


Figure 2. Foam Dyeing Set-up in Laboratory

Instrumentation was added to measure oxygen content, temperature, humidity, and yarn moisture content, so that the impacts of variations in these could be evaluated. Interaction effects among these variables and with variations in the foam blow ratio, wet pick-up, and running speed are also factors to be considered.

Nitrogen gas was used to make the foam dye mixes, chosen as the most cost-effective and safe substitution for air. Its main drawback is its propensity to cool and dry the environment within the application chambers; however, offsetting these impacts was not difficult.

## Results

The prototype system has been sufficient to validate the stipulated hypotheses going into the project, while revealing several modifications that will be needed to produce a sufficiently versatile commercial foam dyeing system. Following are some of the major results obtained.

- Effective dyeing is obtained on yarns taken from spinning machines. Thus, the costly and water-intensive pre-treatment of yarns required with the traditional technology may be eliminated.
- Dyeing is efficient at speeds between 20 and 100 yards per minute (ypm); therefore, since the yarn handling system does not run faster than 100 ypm, a maximum running speed is not yet known.
- Keeping the oxygen content below a threshold is critical to the shade and dye fastness. This is true in both the application chamber and the kinetic chamber.
- Effective dyeing on most yarns may be achieved at moisture levels that leave the yarn just damp to the touch, which require a small amount of heat to dry.
- The flexibility of the system enables control of dye placement within the yarns to greater extremes than is now possible with conventional technology.
- The system also allows changing variables like dye concentration, blow ratio, and wet pick-up in real time, without stopping the dyeing run.
- Both pre- and post-washing is eliminated from the dyeing process. Dyed yarns are immediately available as inputs for downstream processes in fabric formation.
- The elimination of dye baths and the consistency of the applicator/yarn interface have potential to eliminate shade differences when going scaling up from a smaller sample fabric width to full-width fabric.
- The system leaves no waste to collect; zero discharge is a feature of the process.
- Due to the reduction of oxidized surface indigo, colour fastness is superior to existing indigo dyeing technology. Accompanying this result is less tendency to detect a reddish, metallic-like tint (“bronziness”) to the fabrics.
- While the existing prototype dyeing system has limited capability to test the impacts of multiple passes through the application chamber, preliminary results offer optimism that very dark shades (called “black” shades) can be achieved.

Beyond the improvements in coloration, the gains in commercial operational efficiency, when compared with current dye-bath technology, include the following:

- Elimination of the difficult, labour-intensive re-beaming of ropes by doffing either to a take-up beam or directly onto yarn packages.
- Reduction of machine stops from broken yarns. There is also a small amount of wasted lengths of yarn compared to current production when a machine stop is necessary. (This alleviates one of the major drawbacks of slasher-type

dyeing, which currently threatens losses of several million metres when machine stops are required.)

- Elimination of water treatment costs associated with indigo dyeing.
- Elimination of large dye bath circulating pumps, dye bath transfer pumps, and squeeze drive motors, along with their energy and maintenance requirements.
- Elimination of the cost and floor space and maintenance of multitudes of guiding rollers, dye bath storage tanks, and dye vats.
- Elimination of the many different dye baths required for different aesthetic effects, which must be either wasted or re-tested and re-balanced before these can be reused.
- Elimination of the need for rinsing of yarns prior to weaving.
- Elimination of the stress on the yarns from passing over many rollers currently used in preparation, dyeing, oxidizing, washing and drying; this would improve yarn elongation and strength, thereby increasing throughput in the sizing and weaving processes.
- Cost-effective changeovers to different shades (due to real-time changes in dyeing parameters, smaller machine sizes, well-organized yarn sheets, low moisture content, etc.).
- Enclosed-system control of the dyeing environment.

Taken together, the various improvements provide superior operational precision, flexibility, and simplicity; thereby reducing the time required to develop and deliver new denim fabrics.

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The intellectual property derived from this project was acquired by Indigo Mill Designs LLP. Patent applications have been filed internationally. The company continues to facilitate the research and development and is working to bring the technology to commercial status. Inquiries may be made to:

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