Indigo:
Sources, processes and possibilities for bioregional blue

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November 2017
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This report was completed with funding generously provided by the Jena and Michael King Foundation as part of Fibershed’s True Blue project. It is one project of many that support Fibershed’s larger mission:

“Fibershed develops regional and regenerative fiber systems on behalf of independent working producers, by expanding opportunities to implement carbon farming, forming catalytic foundations to rebuild regional manufacturing, and through connecting end-users to farms and ranches through public education.”

In this report we present the various sources of blue dye and of indigo, and motivate the use of plant-based indigo in particular. We also identify the limitations of natural dyes like indigo and the need for larger cultural and systemic shifts.

The ideal indigo dye production system would be a closed-loop system that moves from soil to dye to textiles and back to soil. The indigo process has three basic steps: planting, harvesting, and dye extraction. In this document, we provide an overview of each, and detailed explorations are given in two separate documents that will be available through Fibershed by the end of 2017.

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This report is based on a literature review of academic research, natural dye books, online content, and personal interviews. It benefited greatly from conversations with (and the generosity of) many skilled artisans and natural dyers, including Rowland Ricketts, Jane Palmer, and Kori Hargreaves.
Dyeing is a major component of the bioregional textile systems Fibershed aims to support. In the standard industrial processes, this step is also one of the most polluting. According to the World Bank, 17-20% of industrial water pollution comes from textile dyeing and treatment. Seventy-two toxic chemicals in our water come solely from textile dyeing, of which 30 cannot be removed.

Blue dyes have a long and rich history, and blue represents one of the most striking and challenging colors available from nature’s palette. Humans have used indigo in particular for thousands of years and throughout the world. In fact, 6,000-year old cotton textiles were recently discovered in a coastal area of Peru that were dyed in indigo, and these represent perhaps the oldest known examples of cloth dyeing in the world.

Indigo has low mammalian toxicity and there is no indication of sensitization in humans after repeated skin applications. In fact, there are long histories of humans using indigo to color their skin and bodies. In ancient Britain, for example, the indigenous tribes were known for painting their bodies and faces with indigo pigment (from the woad plant) before battle. Not only does indigo offer a striking and non-toxic color, it is one of the most practical and durable of natural dyes. Jane Palmer of Noon Dye House in Los Angeles, CA turned from using a range of natural dyes to exclusively indigo for motivations that were both economic and technical. Palmer worked with natural dyes in a production setting for about 15 years, and—while she loves natural dyes in general—most posed huge challenges from a commercial perspective, often fading quickly and having poor light- and wash-fastness and showing great inconsistency between batches.

It was indigo alone that demonstrated advantages in performance, economics, and processing. Among the natural dyes, indigo is notable for high wash and light-resistance. Unlike most other natural dyes, indigo can be used without mordants to fix the dye to textile fibers. Without a mordant bath, Palmer needed less equipment and was able to use significantly less chemical inputs, water, and energy. Indigo was also less susceptible to variations in incoming water, whereas the other natural dyes showed inconsistency with the varied water sources and qualities coming into the dyehouse on a given day. Palmer turned to indigo dye partly because she felt she was wasting so much water tuning the other dyes. It was fortunate that public regard for indigo dye was high, and economic and technical pressures pushed in the same direction.

For its rich history, cultural significance, beauty, low toxicity, performance, practicality, and the opportunity to reimagine a highly polluting industry, natural indigo presents a great opportunity, and supporting a local industry is a compelling goal for Fibershed and those who share its values.

2. Los Angeles Times (2017) “6,000-year-old fabric reveals Peruvians were dyeing textiles with indigo long before Egyptians”
5. Personal conversation
Sources of blue

While there are multiple modern methods of achieving blue colors in textiles—most notably fiber-reactive dyes such as those based on dichlorotriazine (e.g. Procion MX) or anthraquinone (e.g. Remazol Blue R)—natural indigo is the only widely used blue dye that both is non-toxic and can be produced from renewable resources.

Unlike fiber-reactive dyes, both natural and synthetic indigo function by adhering only to the surface of textile fibers. The pigment can rub off of the fibers (known as “crocking”). While this could be seen as a downside, it is also the property that contributes to the favorable appearance of faded denim and is seen as a desirable feature for many textile products.

Sources of indigo

Indigo accounts for roughly 3% of all dyes used globally today and about 15% of the dyes used for cotton in particular.6

There are ongoing projects of innovation in the dyeing industry, including non-indigo blue pigments from natural microbial sources (from groups like France’s Pili.bio) as well as developments in structural color, which takes advantage of nanostructured surfaces to scatter light and produce color (as can be seen in the colorful feathers of a peacock). While these developments are certainly promising, none are market-ready, and natural indigo remains the most viable and safe method available.

Chemistry of Indigo Extraction

The chemical precursor to indigo in plants is indican, a glycoside. It exists throughout the plant but is concentrated almost exclusively in the vacuoles of the leaves.9 Although indican concentration can reach as much as a few percent of the wet weight of the leaves, we still do not fully understand its biological function for the plant.

The extraction and subsequent hydrolysis of indican (achieved through heat and/or fermentation in water) cleaves the indican into two parts, resulting in β-D-glucose and indoxyl. The hydrolysis is achieved via enzymes naturally present in the leaves as well as by the actions of microbes that likely seek the β-D-glucose as food.

The interaction of indoxyl with a mild oxidizing agent (such as atmospheric oxygen) under conditions of high pH yields the blue pigment indigotin (the active pigment in indigo dye).

Indigotin is only one of many oxidation products of indoxyl. Under non-ideal conditions, others—such as isatin and indirubin—can be formed, contributing to lower purity dyestuffs with lower quantities of the desired indigotin pigment.10

The annual world production of indigo dye was about 80,000 tonnes in 2010.7 Worldwide production of denim, the fabric from which jeans are made, uses more than 95% of that production.8

While indigo was originally produced from natural sources, there are now multiple methods to generate the molecule, all of which can and have been used for dye production.

There are three general approaches to producing indigo: natural plant-based methods, synthetic petroleum-based methods, and newer microbial-based methods using genetically-modified microorganisms.

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8 Wolf, L. (2011) “Blue Jeans: Making the iconic pants requires both color-addition and color-removal chemistry”
9 Minami, Y. et al. (2000) “Tissue and Intracellular Localization of Indican and the Purification and Characterization of Indican Synthase from Indigo Plants”
PLANT-BASED INDIGO
Plant-based methods involve the extraction and processing of naturally-occurring indican in plants and include the compost process and the water extraction process, which are discussed later on.

Currently natural indigo pigment represents less than one percent of the indigo dye produced and used worldwide. The main uses are as colorant for beauty products like hair dye and for designer denim and artisanal textile products. It is commonly available at purities of 20-50% at a price of $20-70/lb. Production is concentrated in Japan, and some US domestic production is underway—most notably through Stony Creek Colors in Tennessee, who currently contracts with 16 farmers to cultivate about 180 acres of indigo. For compost, production is centered in Japan, which has a long-standing tradition of producing indigo compost known as “sukumo”, and where production reached about sixty tons per year between 1985 and 2009.

SYNTHETIC INDIGO
Synthetic methods include the Heumann method (developed in 1897) and the Pfleger method (developed in 1901), as well as other methods developed more recently. All of these synthetic methods use crude oil as a feedstock and employ a long series of sophisticated petrochemical techniques.

Synthetic indigo is available in powder, granule, or liquid (solution) form. Liquid solution tends to be most readily available in 20%, 30%, and 40% pigment content. Powdered and granule forms tend to be most readily available at 94-100% purity.

The price of synthetic indigo ranges from about $2/lb to $5/lb. The majority is produced in China, with other suppliers in Europe and other parts of Asia. A full 40% of the market is controlled by BASF, with other major players including Japan’s Mitsui and England’s Imperial Chemical Industries.

MICROBIAL INDIGO
Microbial methods involve inserting the genes for indican production from plants into microorganisms that can then be cultured and concentrated. The process has been employed at the lab scale for decades. One recent method is that developed by Professor John Dueber and his lab at UC Berkeley.

Using sugar feedstocks and genetically-modified E. coli, the Dueber Lab grows cultures that can produce significant quantities of indigo. Dueber reports indigo production from his cultures of 0.4 g/L and claims that production of 4 g/L is easily imaginable in an optimized process.

As one point of comparison, trials in Central Italy in 2001 and 2002 showed potential indigo pigment yields for Persicaria tinctoria (syn. Polygonum tinctorium) of up to 291 lbs/acre. This 291 lbs of indigo would take approximately 13,200 or 132,000 liters of Dueber’s liquid culture to produce—a liquid volume equivalent to that of a cube about 8 or 16 feet to a side.

It is unclear at this time how the full life-cycle of microbial indigo affects the ultimate environmental impact of the technology. For such an analysis, the significant land and resources required to grow and process sugar feedstocks would need to be taken into account.

The technology of microbially-produced indigo is not new, and its story demonstrates that the synthetic indigo industry is a force to be reckoned with. In the early 2000s, Bay Area biotechnology company Genencor partnered with Levi’s to produce 400,000 yards of microbial indigo-dyed fabric at near-market rates. Despite this success, the technology never took off, as Levi’s synthetic indigo producer in China promptly slashed prices and pushed Genencor out of the market.

There are possibilities for directly dyeing textiles with indoxyl, a precursor to indigo in the water extraction process. In fact, home-dyers may be familiar with this process as “green leaf dyeing”, a process in which green leaves are blended and used immediately to dye textiles a light blue. As the Dueber Lab has demonstrated, it is possible to achieve dark blue colors with this process using either plant-sourced or microbially-sourced indican.

The process allows for in-situ indigo formation on textiles, bypassing the additives required to separately precipitate indigo in dye production only to later re-dissolve it in the dyebath. Indoxyl, however, is much less stable than indigo, and until a process for controlling and sustaining indoxyl in solution is developed, the process cannot be employed at scale.
Why plant-based indigo?

Nearly all indigo in production today comes not from renewable resources, but as a product of the petrochemical industry, with concomitant toxicological and environmental concerns.

In general, the synthetic processes of indigo production are complex, far-reaching, and difficult to track. See the image below for an overview of the Pfleger method, one of the major methods currently employed.

While no study has fully characterized the human and environmental consequences of synthetic indigo production, an understanding of the process certainly raises concern. For example, one of the many intermediate products in the process above is aniline, a chemical that is toxic if ingested, inhaled, or touched that inhibits the ability of blood to carry oxygen and is fatal in high exposures.

In contrast, natural indigo can be grown in an entirely sustainable manner. It can be grown in a way that promotes soil health and soil carbon sequestration, and no dangerous chemicals or processes are required. Even the waste-products can be employed beneficially: After the pigment has been extracted, the plant residue can be composted and used as a fertilizer and the remaining water can be reused to irrigate crops.

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**Synthetic Indigo Production (Pfleger Method)**

**Crude oil**

- Catalytic reforming
- Toluene hydrodealkylation
- Toluene disproportionation
- And/or steam cracking

**Benzene**

- Nitric acid
- Sulfuric acid
- Metal catalysts
- Heat

**Aniline**

**n-Phenyglycine**

- Formaldehyde
- Hydrogen cyanide
- Sodium hydroxide

**Indigo**

- Molten potassium hydroxide
- Sodium hydroxide
- and soda ash

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Information and images from https://www.wikipedia.org/

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19 Information and images from https://www.wikipedia.org/
Sources of plant-based indigo

There are many plant sources for indigo, including India’s *Indigofera tinctoria*, Central America’s *Indigofera suffruticosa*, and the woad plant, *Isatis tinctoria*, used extensively in Europe.

[Image of Indigofera tinctoria](https://www.denimhunters.com/how-denim-is-made-indigo-dyeing/)

[Image of Woad, Isatis tinctoria](https://en.wikipedia.org/wiki/Isatis_tinctoria)
The temperate United States is best suited for a plant known as dyer’s knotweed or *Persicaria tinctoria*, a native to Japan, and the True Blue project focuses on the cultivation and use of this plant.

*Persicaria tinctoria* has multiple varietals, such as “senbon” with red flowers and rounded leaves, and “kojoko” with white flowers and pointed leaves.

**INDIGO CONTENT**

The *Handbook of Natural Colorants* includes a literature review of the indigo content of various plant sources. It reports the fresh-leaf indigo content in *Persicaria tinctoria* from five separate studies, giving an average yield of 0.57% and ranging from 0.13% to 1.24%. The same report presents six studies on *Isatis* species, giving indigo contents ranging from 0.02% to 0.80% with an average of 0.19%.

These values refer only to the indigo content of the plant leaves, where indigo is most highly concentrated. The stems of the plant have much lower indigo contents, and their inclusion in dye extraction processes would result in lower yields than those reported above.

*Indigofera* species are generally recognized to be the highest in indigo content, and exceptional samples of *Indigofera tinctoria* might yield indigo to values of 3.5% of the dry leaf weight. There have been, however, no published studies using modern techniques to determine indican content of *Indigofera* leaves, and the absence of such information makes it difficult to compare the efficiency of *Indigofera* to that of other crops.

**PURITY**

Vuorema provides a helpful overview of the considerations in indigo purity:

“Natural indigo contains besides indigo, impurities such as indirubin, indigo-brown, indigo gluten and mineral matter. The indigo purity has been reported to be for woad indigo 20-40%, for *P. tinctorium* up to 12% and for *Indigofera* indigo the highest from 50 up to 77%. There is also the question of the efficiency of the extraction, the theoretical yield of the indigo formation from indoxyl molecules have been discovered to be approximately 60%. So 40% of the indoxyl is lost during the process to impurities such as isatin and indirubin and other by-products of the reaction.”

Other sources suggest different purities for indigo powder from *Persicaria tinctoria*. For example, the purity of natural indigo powder sold by Stony Creek Colors is advertised as approximately 40%, and purities claimed by other producers can range from about 20% to 50%.

These values are far lower than those achieved with synthetic indigo, with purities routinely exceeding 90%.

Consistency is also a key consideration. For most large-scale producers, inconsistency between batches of indigo will represent a problem for quality control. For some smaller-scale producers or artisans, however, the complexities of the natural dye and the slight variation between batches can actually be desirable and presented as features of the product line.

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[22 Personal correspondence from Rowland Ricketts]


Limitations of natural indigo

Even naturally-sourced indigo presents problems as an industrial-scale dyestuff, including the large amounts of land and water employed in the process and the production of highly alkaline and dye-laden wastewater.

Plant-based natural indigo makes up less than one percent of the indigo dye currently produced worldwide. If the total annual production of indigo dye was sourced from plants, it would require something on the order 2 million acres of cultivated land. That area is equivalent to the area of a square roughly 56 miles to a side.

As argued in a 2015 article in The Guardian, natural dyes in general have serious consequences in industrial-scale production, and alternative solutions and innovations are needed if we are to meet the demands of industry. The article quotes Simran Lal, the CEO of India’s GoodEarth luxury clothing brand, and Phil Patterson, a UK-based textile consultant:

“[GoodEarth’s Lal argues that] natural fabrics and dyes are a luxury product, not suitable for mass consumerism. ‘Given the complexities of creating natural dyes and the resources required, such as water and land against the vast commercial markets across the globe, it’s not possible to use only natural dyes.’

Consequently, GoodEarth employs both natural and synthetic dyes in its clothing and linen collections.

Ultimately, the problem hinges on a larger issue. ‘The world has gone consumption-mad,’ Patterson says. ‘As a result there are enormous amounts of textiles produced.’ He argues that consuming less is the only way to make an impact in the long run.”

http://www.noorism.com/single-post/2015/12/05/DIRTY-DENIM

Based on indigo process modeling that is presented in an upcoming Fibershed report on indigo dye extraction

The Guardian (2015) “Natural dyes v synthetic: Which is more sustainable?”
The ideal indigo dye system would be a closed-loop system that moves from soil to dye to textiles and back to soil. The process of producing indigo dye has three key steps: planting, harvesting, and dye extraction. Dye extraction can be carried out using either the water extraction method or the compost method. Planting, harvesting, and dye extraction are explored in detail in two separate documents, and here we provide an overview of each.

**Planting**

While indigo can be planted directly in the field, it is usually desirable based on climate and yield to start seedlings in a greenhouse and to later transplant them into the field. Such seedlings can be started by broadcasting seeds onto open beds or by planting into flats. While the broadcasting method is the simplest, the seedlings produced are generally only transplantable by hand. Starting seedlings in flats allows for greater flexibility and the possibility of automation in the transplanting step.

**Persicaria tinctoria** likes a neutral, well-drained soil, full sun, and weekly watering. In the Northern California fibershed, indigo can be transplanted as early as April after threat of frost has passed.

Transplanting can be completed with varying levels of automation, and the ideal solution for a given producer will depend on the size of the cultivated area and the availability of capital.
For home-scale to scales of hundreds of acres, methods range from transplanting by hand to using hand-held tools, walk-behind machines, semi-automated tractor-mounted machines, and fully-automated tractor-mounted systems. We give recommendations and an overview of planting methods in a separate document.

**Harvesting**

*Persicaria tinctoria* is typically harvested twice per year. In the Northern California fibershed, the first harvest occurs around July. The second harvest occurs around August, and the plant is then allowed to go to seed.

Timing is very important, as the indigo concentration in the plants can vary greatly throughout the year (as does, of course, the size of the plants themselves). As seen below, indigo concentration over time in *Persicaria tinctoria* is related to the amount of light available to the plants as they grow, known as photosynthetically active radiation.

There is an opportunity to optimize indigo yield by varying the number and timing of harvests, and the ideal parameters will depend on location and the variety of indigo used. For northern California, we currently recommend harvesting twice per year, once in early to mid-July and once in mid- to late-August. For the first harvest, cut the plants when they are about 1-2 feet tall, cutting the stems several nodes up from the ground. The plants will resprout from those nodes for the second harvest, which should be completed before or shortly after the plants flower. A third harvest may be possible in early to mid-October, but indigo concentration may be low by this point.

Depending on scale of production and available capital, the ideal harvesting method can range from hand-harvesting to using walk-behind harvesting machines or tractor-mounted systems. We give recommendations and an overview of harvesting methods in a separate document.

**Dye Production**

The two main methods for producing indigo from plants are the water extraction process and the compost process. Both methods serve to convert naturally-occurring indican in the plant cells into indigotin pigment and to concentrate this pigment into a usable dyestuff. The water extraction method accomplishes this in the liquid state, and the compost method accomplishes this in the solid state.

We have written a separate report that focuses on the water extraction and compost processes in detail. On the following page, we present a brief overview.

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30 Angelini, L. et al. (2004) “Environmental factors affecting productivity, indican content, and indigo yield in Polygonum tinctorium Ait., a subtropical crop grown under temperate conditions”
WATER EXTRACTION METHOD

The water extraction process is a liquid-state extraction process that yields a powdered pigment with higher indigotin purity than indigo compost. Once dry, the pigment is compact, relatively homogeneous, predictable, and shelf-stable.

The basic steps of water extraction include extraction, alkalization, oxidation, and filtration. In the extraction step, the plants are submerged in water then heated and/or fermented to pull the indigo precursor indican out of the plant cells and into solution. Once in solution, the indican undergoes hydrolysis (via enzymatic and/or microbial actions) and cleaves into indoxyl and a sugar. After extraction is complete, the spent plant material is separated and set aside. The remaining indoxyl-laden liquid is first alkalized (given a basic pH) then oxidized (usually through aeration), producing indigotin. The insoluble indigotin precipitates, settles, and is filtered, optionally rinsed, and finally dried to produce indigo powder.

COMPOST METHOD

The compost process uses composting to concentrate (rather than extract) the pigment found in indigo plants. Japan has a long-standing tradition of producing a composted indigo dyestuff known as “sukumo.” In this tradition, indigo plants (Persicaria tinctoria) are first harvested and dried. The leaves are then separated and composted for approximately 100 days, with frequent turning of the pile. After composting is complete, the remaining material (“sukumo”) is dried and bagged, ready for use in traditional dyeing vats.

In the Japanese tradition, indigo plants are harvested by hand and then laid out to dry, occasionally being moved to ensure even drying.

Once the leaves are dry but the stems are still soft and flexible, people stomp on the drying plants, crushing the dry leaves into small pieces and separating them from the stems. The stems are then composted and the leaves collected.

The dry indigo leaves are piled on a “compost pad” (a particular floor construction that allows for good moisture drainage). The pile must contain at least about 450 lbs of dried leaves for effective heat-retention and composting, and in practice piles can weigh many thousands of pounds.

Over a period of 100 days the compost pile is carefully turned and mixed, with water and various amendments being added to the pile to keep it biologically active. Over time this process reduces the mass and volume of the leaves by roughly 75%, concentrating the indigo. Processes other than simple concentration are likely occurring due to microorganisms and their byproducts interacting with the indigo compounds, but this is not well understood.
THE PROCESSES OF DYEING WITH INDIGO POWDER AND COMPOST

Both indigo powder and compost contain indigotin, the blue pigment in indigo dye. When using either indigo powder or compost for dyeing, the water-insoluble indigo molecules are chemically reduced in a dye bath and converted into a green-colored water-soluble form known as leuco-indigo. This dissolved leuco-indigo saturates textiles when they are dipped into the vat, and when the textile is removed and exposed to atmospheric oxygen the leuco-indigo oxidizes again to water-insoluble indigotin, and the pigment adheres to the fibers. As the leuco-indigo oxidizes into indigotin, the color of the textile changes dramatically from greenish to indigo blue.

Reduction of indigo into leuco-indigo can be achieved in a number of ways, including biological and chemical methods. Japanese sukumo is typically reduced biologically in large vats using a traditional process that involves water, wood ash, limestone, and wheat germ. Artist and professional indigo dyer Rowland Ricketts trained in indigo growing and dyeing in Japan and practices the Sukumo method in his work. He provides an excellent illustrated overview of the process on his website:
http://www.rickettsindigo.com

Indigo powder can be reduced with natural materials33, 34 such as fructose, ferrous sulfate, henna, osage, and madder, or with synthetic compounds such as sodium hydrosulfite and thiourea dioxide.

We do not address specific methods for indigo dyeing in this document.

For more resources, we recommend the following books, online materials, and teachers.

Books
The Modern Natural Dyer
by Kristine Vejar

Natural Color
by Sasha Duerr

Harvesting Color
by Rebecca Burgess

A Handbook of Indigo Dyeing
by Vivian Prideaux

32 Bechtold, T; Mussak, R (2009) “Handbook of Natural Colorants”
34 http://www.aurorasilk.com/tutorials/how_to_natural_indigo_dye_vat.html
For its rich history, cultural significance, beauty, low toxicity, performance, practicality, and the opportunity to reimagine a highly polluting industry, natural indigo presents a great opportunity, and supporting a local industry is a compelling goal for Fibershed and those who share its values.

The indigo process has three basic steps: planting, harvesting, and dye extraction. In this document, we provided an overview of each, and detailed explorations are given in two forthcoming documents that will be available through Fibershed by late-summer 2017.

In this document, we also presented the various sources of blue dye and of indigo, and motivate the use of plant-based indigo in particular. We identified the limitations of natural dyes like indigo and highlighted the need for larger cultural and systemic shifts.

We believe plant-based indigo is one of the best available options for regionally focused dye projects. We view natural dye farming as an important facet of integrated food and fiber farming projects that can support local economies. To have the desired effect on culture and industry, however, we believe natural indigo needs to be part of a larger symbolic and strategic program that recognizes direct competition with the vast synthetic indigo industry is not possible.

We recommend pairing regional natural dye projects with efforts to shift culture and our patterns of consumption through building appreciation, respect, and understanding for the resources, skill and labor required to make plant-based blue a reality for modern wearers.

Conclusions