# Product Quality in the Natural Indigo Production from *Polygonum tinctorium L.*

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## Abstract

전통 쪽 염료인 니람 제조원리를 바탕으로 산업적인 쪽 염료 제조공정에 대한 제안과 함께 이와 관련한 몇몇 공정변수를 포함한다.

## 1. Introduction

The Niram method is one of the traditional processes in Korea. In this method, plant glycoside (indican) is extracted by steeping plant materials for 1-3 days. The powder of oyster shell is added to the extract while stirring manually for the introduction of air oxygen as much as possible until blue color developed, and then left it overnight. The sediment is collected and filtered through a cotton fabric. The quality and quantity of the dye are variable because the conditions for extraction and subsequent conversion into indigo are hard to control. The objective of this study is to optimize the dye quality by controlling process variables.

## 2. Experimental

### 2.1 Preparation of indigo plant and extraction of indigo dye

Indigo leaves (50 g) harvested were steeped in distilled water (1 L) at 25-26℃. Leaves were held under water to exclude air, thus maintaining anaerobic conditions. The extraction was made by maintaining leaves in distilled water at various time and pH. After filtering the extract, $Ca(OH)_2$ was added with stirring for 20 min using a homogenizer, and left it overnight for sedimentation. Supernatant was siphoned off and then the slurry (sediment) was dried in a laboratory dryer at 50℃ and weighed. The dried sediment was finely ground and used for analytical tests and dyeing process.

### 2.2 Large scale extraction of indigo dye

Into a 1000 L tank, water of 27℃ was poured over the plants with leaves and stems (80 Kg, 10 L of water to 1 Kg of plant materials). The steeped plants were weighted down with 6 stone-blocks (weighing 5 Kg each)

to hold plants under water to exclude air for maintaining anaerobic condition. After steeping 2.5 days, the extract (790 L) was pumped into three settling tanks and added different amount of Ca(OH)2 (2.0, 2.25, and 2.5 g/L) in each tank respectively, and aerated for 30 min using a compressor to help oxidation of indigo precursors and precipitation of indigo. The indigo was settled down for 12 hrs and the supernatant was removed. The sediment was collected, filtered, and dried in a vacuum oven at 50℃, the amount of crude indigo dye was weighed and pulverized for analytical tests and dyeing experiments.

### 2.3 Indigo and indirubin quantification

Indigo and indirubin contents were determined according Liau et al.[1-3]. HPLC analysis was carried out on a Agilent 1200liquid chromatography system (Agilent technologies Inc., Waldbronn, Germany) equipped with two pumps, UV detector and Rheodyne injector (50L loop).

## 3. Results and Discussion

Total mass of crude dye obtained was dependent on extraction time and the amount of $Ca(OH)_2$. Increase of the amount of crude dye within 1-2.5 days of extraction was observed with the extraction time. Extraction time of longer than 2.5 days caused a slight decrease in the amount of crude dye. The quantity of dye extracted was increased with the amount of $Ca(OH)_2$. In lab scale results, optimum extraction condition was considered to be 2.5 days for extraction time and 2.0-2.5 g/L of $Ca(OH)_2$ for precipitating indigo dye.

Based on the lab scale extraction, large scale extraction was done for 2.5 days and the amount of $Ca(OH)_2$ was applied with 2.0, 2.25, and 2.5 g/L. Even though stems were included during extraction, indigo content was comparable to that of lab scale production using leaves only. The harvest time is important to get high dye quality. Additionally, higher indirubin content in the large scale production was obtained.

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## References

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