Faculty of Applied and Creative Arts

EXPERIMENTATION OF INDIGO DYEING USING DIFFERENT NATURAL REDUCING AGENTS TO PRODUCE APPAREL PRODUCTS

Liew Xiao Xian

Bachelor of Applied Arts with Honours
(Design Technology)
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LIEW XIAO XIAN

This project is submitted in partial fulfillment of
the requirements for the degree of Bachelor of Applied Arts with Honors
(Design Technology)

Faculty of Applied and Creative Arts
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2018

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ABSTRACT

Indigo is the world's only natural blue dye. At the industry level, indigo dye reduced in high alkalinity using sodium dithionite is of significant importance. However, the process is environmentally unfriendly. This study focuses on the experimentation of indigo dyeing using different natural reducing agents. Experiments using various parameters are carried out and different fruits are tested for the reduction potential with the recipe proportion of 1:2:3 (Quantity of indigo powder: calcium hydroxide: reducing agent). The results revealed that different blue shades are able to be produced using different reducing agent, while crystalline fructose is the strongest reducing agent. The finding from the experiments has resulted in the creation of a sustainable indigo project in producing apparel products. A collection of women Ready-To-Wear fashion is produced at the end of this research. This research showed that the use of different reducing agents induced a more ecological way of indigo dyeing.

Keywords: Indigo, Natural dye, Natural reducing agent, Apparel products.

ABSTRAK

Indigo merupakan satu-satunya pewarna biru semula jadi di dunia. Di peringkat industri, pewarna indigo yang direduksikan dalam kealkalian tinggi menggunakan sodium dithionite adalah penting. Bagaimanapun, proses tersebut tidak mesra alam. Kajian ini memberi tumpuan kepada experimentasi pewarnaan indigo dengan menggunakan agen reduksi semula jadi yang berbeza. Eksperimen menggunakan pelbagai parameter dan buah-buahan yang berbeza diuji untuk potensi reduksi dengan nisbah resepi 1: 2: 3 (Kuantiti serbuk indigo: kalsium hidroksida: mengurangkan agen). Hasil kajisan menunjukkan bahawa warna biru yang berbeza dapat dihasilkan menggunakan agen reduksi yang berbeza, sementara fruktosa kristal adalah agen pengurang yang paling kuat. Dapatan kajian dari eksperimen ini telah menghasilkan penciptaan projek indigo yang mampan dalam menghasilkan produk pakaian. Koleksi fesyen wanita Sedia-Untuk-Pakai dihasilkan pada akhir kajian ini. Kajian ini menunjukkan bahawa penggunaan agen reduksi yang berlainan memperkenalkan cara yang lebih ekologi untuk pewarnaan indigo.

Kata kunci: Indigo, Pewarna semula jadi, Agen reduksi semula jadi, produk pakaian.

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CHAPTER 1 INTRODUCTION

1.0 Introduction

This study is about indigo dyeing using different reducing agents. Indigo is the world's only natural blue dye. It is called by so many names, such as ai (Japan), landian (China), khram (Thailand), chàm (Laos and Vietnam), nila (India), gara (Africa), or añil (Central America). It is one of the best known natural colouring agents and was the only source of the dye until the late 19^{th} century.

The indigo blue is obtained from various indigo bearing plants, such as *Indigofera tinctoria, Polygonum tinctorium* (also known as Japanese Indigo), *Nereum tinctorium and Isatis tinctoria.* This shrub grows wild and is cultivated in tropical areas throughout the world. The plant produces a colourless glucose based-substance called indicant which is a precursor to indigo dye. The dye is extracted from the processing of the plant's leaves. The leaves are soaked in water and fermented to convert the glycoside indicant to the blue dye indigotin (the dyeing component of indigo).

Indigo belongs to a class of dyes called vat dyes. Vat dye molecules are insoluble in water and in their original form and have no affinity for fiber. They must be reduced (to have their oxygen removed) to form a soluble dyestuff that can be bind to the fiber. Therefore, indigotin must be reduced to a water soluble form known as indigo white. There are different ways to reduce the indigotin, the most common one being chemically reduced by using sodium hydrosulphite, Na_2SO_3 and thiourea dioxide, $CH_4N_2O_2S$. A molecular combination occurs (oxidation process) when fiber is dipped to an indigo vat (amber or yellowish-green colour) and then removed. The indigo white then is oxidized back to the insoluble blue state, where it will remain in relative permanence.

In the colouration of cellulose fiber, indigo vat dye represents a relatively important part of the dyestuff market which is about 7% of the world dyes consumption. The consumption of indigo and other vat dyes reaches about 33 million kg annually (Roessler & Jin, 2003). And the reduction of indigo to indigo-white represents an important type of industrial process which is operated worldwide on a considerable scale (Roessler & Crettenand, 2004). The popularity of blue jeans is the reason behind the huge consumption of indigo dye. Since the 60's, enormous developments comprised of changes in conventional reduction and application techniques, modification in design of machineries and ways to enhance indigo uptake has been proposed due to high demand of jeans (Chakraborty,2003). However, the conventional method in the indigo dyeing has reduced the good quality natural indigo dyed products. Therefore, this research has significant importance in increasing the quality and beauty of natural indigo.

There have been many alternative methods developed in indigo dyeing to encourage a cleaner textile process. In this study of indigo dyeing, different reducing agents will be tested in the indigo reduction process to produce various shades of indigo blues.

This study is important to grow people interest towards a more eco-friendly indigo dyeing. It is hoped that the value of natural indigo can be enhanced through this research and help to raise people's appreciation towards the depth and beauty of indigo blues. The new knowledge of the indigo dyeing using organic indigo vats may give great understanding of indigo reduction by natural reducing agent as well as to offer advantages to the environment which will help to reduce uses of chemical reducing agent and synthetic indigo in the production of indigo textiles.

1.1 Background study

Natural indigo is made through a non-toxic fermentation of the indigo plant. Depending on the variety of indigo available locally, some artisans and dyers will make a dye vat directly from the plant leaves. The indigo extract may be produced as lumps or fine powder.

In modern indigo dyeing process, the indigo dye is chemically reduced in a high alkaline solution (pH about 13), which the reduction process is considered ecologically unfavourable (Roessler, 2004). As a result, studies are being carried out to replace sodium dithionite, $Na_2S_2O_4$ (chemical reagent used in indigo reduction). One of the organic biodegradable studies of "Comparative Study on Traditional Indigo Dyeing onto Cotton Fabric Using Ripe Banana and Sodium Dithionite as Reducing Agents" published in Asian Journal Chemistry showed that ripe banana can produce dyeing results similar to sodium dithionite. The obtained result from the study showed that there are more to discover about indigo dyeing by using environmentally friendly reducer.

Also, there has been an increasing interest about organic vat in indigo dye. The organic vat is developed based on the traditional indigo vats of Morocco, India and Provence, which relies on the chemical reactions between a mineral alkali and a natural reducing agent to remove excess oxygen. These natural reducing agents include natural sugars from dried and fresh fruits, medicinal plants, minerals, and flavanoids (Maiwa Foundation, 2013). There are countless variations on this vat such as fruit, henna, fructose, and ferrous vat. In spite of this information, it is not known that to what extend these fruits can produce various shades of blue. Hence, this paper presents the deeper understanding about use of different organic materials as natural reducing agents in indigo dyeing.

1.2 Research Problem

a. **The use of strong chemical reducing agent (sodium dithionite, $Na_2S_2O_4$) in indigo reduction is ecologically unfriendly**

Indigo has been extensively used in textile and fashion industry. In most industrial indigo dyeing process, sodium dithionite ($Na_2S_2O_4$) is commonly used as the reducing agent in the vat dye reduction due to its economic properties (Schlüter, 1990). However, the use of sodium dithionite as reducing agent produces environmentally harmful decomposition by product such as sulphite (SO_3^{2-}), sulphate (SO_4^{2-}), thiosulphate ions ($S_2O_3^{2-}$), and toxic sulphur (Aspland, 1992). These toxic and corrosive chemical wastes give rise to various waste water disposal problems. According to Božič and Kokol (2008), the excess discharge of sodium dithionite ($Na_2S_2O_4$) affects the aerobic processes in wastewater treatment. This may cause toxic hydrogen sulphite (H_2S) to form anaerobically from the sulphate present in the waste water.

Sodium dithionite ($Na_2S_2O_4$) is easily to be oxidized by atmospheric oxygen, and its alkaline solutions stability decreases with the increase of temperature (Camacho, Jiménez & Fernández, 1997). As a consequence, over stoichiometric requirements of the reduction process, great amounts of sodium dithionite and sodium hydroxide (NaOH) are needed to sustain the dye bath. The worst thing about using sodium dithionite is it cannot be recycled from the waste waters and used again in the indigo dyeing process (Bechtold, Burtscher, Amann & Bobleter, 1992). Subsequently, this will resulted in large chemical disposal which will give adverse effect on the environment.

b. **Health hazards of using chemical reducing agent in indigo dyeing**

The storage of sodium dithionite is associated with issues like risk of fire and health hazards. Apart from sodium dithionite, thiourea dioxide ($CH_4N_2O_2S$) is also used as reducing agent in indigo vat. It is not only an excellent substitution for sodium dithionite, but also environmentally friendly and safer to use, has a greater strength and better shelf life. However, thiourea dioxide is considered hazardous by the 2012 OSHA Hazard Communication Standard (29 CFR 1910.1200). It can cause skin, eyes and respiratory irritation without adequate safety precaution. Moreover, prolonged or repeated exposure to thiourea dioxide can cause damage to the organs. Artisans, weavers and textile dyers may expose to this health risk.

c. **Alternate methods used to replace chemical reducing agent in indigo reduction is not economical**

Attempts have been made by researchers to eliminate production of inorganic waste from chemical reducing agents. For example, use of sodium borohydride, iron (II) complexes (gluconic acid complexes), biotechnological reduction and electrochemical reduction. Somehow, most of the methods do not seem to be promising due to technical and economic restriction. Meanwhile, the substitution of noxious chemical reducing agent by other reducing agent like α-hydroxy ketones, hydroxyalkyl sulphonate, thiourea are expensive and acrid smell is formed from the condensation products of the alkaline solution.

These existing problems related to indigo dyeing however can be eliminated and minimizing through the utilisation of organic material as natural reducing agent to replace the chemical reducing agent used in the vat dye.

1.3 Research Questions

Among the questions to be answered via this research are:

i. What are the natural resources with reducing properties that suitable to be used to reduce the indigo?

ii. How will different reducing agents produce various colour shades?

iii. What is the natural reducing agent and recipe that produce strongest organic vat dyeing?

1.4 Research Hypothesis

It is possible to produce varieties of indigo colour shades and uniform dyeing by using different natural reducing agent in the indigo reduction process.

1.5 Objectives

The main purpose of this research is to study indigo dyeing by using different reducing agents. Thus, to specify, the objectives of this study are to:

i. Identify the natural reducing agent suitable for indigo dyeing.

ii. Produce different shades of indigo colour using different natural materials contained reducing properties

iii. Determine the strongest natural reducing agent and organic vat dye recipe

iv. Produce indigo dyed products

1.6 Research Scope

This research is mainly focusing on the study of indigo dyeing process specifically in the indigo reduction process by using different organic vat. The scope of the research covers the following aspects:

i. Production of various colour shades using different reducing agent
ii. Methods to optimize indigo reduction
iii. Procedures of indigo dyeing process
iv. Parameters of organic indigo vat dyeing (amount of indigo, base, reducing agent, and volume of water)
v. Manual Light fastness of Indigo

1.7 Research Limitation

i. This research is limited to natural indigo powder obtained from indigo bearing plant (*Indigofera tinctoria)* imported from India only since varieties of indigo are not available locally.
ii. The analysing of depth of shades in the resultant colour from each organic vat sample is restricted to observation only due to the lack of equipment like spectrophotometer.

1.8 Research Importance

The outcome of this research will help to contribute new knowledge and information on indigo reduction by using different organic vat as well as establish a more environmental friendly indigo dyeing process.

As a chemical-intensive industry, textile industry is the most environmentally polluting industry after agriculture (Cao et al., 2014). About 120,000 tons of indigo and other vat dyes are being consumed annually for the colouration of denim and cellulose fibres (Roessler, Crettenand, Dossenbach, Marte & Rys, 2002). It is estimated that consumption of global denim industry will be worth of USD 64.1 billion by 2020 (Muthu, 2017). Thus, this research is important in serving as an initial stage to develop ecologically friendly process for indigo dyeing.

Meanwhile, from the indigo dye aesthetic perspective, the findings of the research will be beneficial to the textile designers, artisans and weavers in producing apparel and furnishing products with different shades of blue using inexpensive and readily available natural reducing agent in the organic vat dye. The appreciation of the beauty of natural indigo can be increased through the making of indigo-based craft textiles.

Unlike South East Asian countries like Thailand, Myanmar and Indonesia, there is less popularity of natural indigo in Malaysia. Thus, this research holds of an importance to promote uses of natural indigo among the locals through the making of indigo-based products by the end of the research. Subsequently, this will be an encouragement for people to support natural indigo production which will generate income for the artisans' community.

1.9 Definition of Terms

Indigo – The plant or the resulting dye that produces a blue colour.

Indicant – A colourless organic compound ($C_{14}H_{17}NO_6$), soluble in water, naturally occurs in *Indigofera* plants.

Indigotin – A dark blue crystalline compound ($C_{16}H_{10}N_2O_2$) that is the main constituent of the indigo dye.

Indigo-white – A pale yellow crystalline compound ($C_{16}H_{12}N_2O_2$) obtained by reduction of indigo and easily changed back to it by oxidation, called also leuco-indigo.

Reduction – Chemical reaction that adds and removes oxygen.

Vat Dye – Water-insoluble dyes that must be reduced in alkali to a soluble state before applying to fabric. They successfully colour cellulosic fibers and are colourfast to light and washing.

Organic Vat- Vat dye made from natural materials.

Organic material- Materials originate from plants that contain organic matter (carbon based compounds).

Fermentation – A chemical breakdown of sugar in the absence of oxygen.

1.10 Conclusion

Indigo is a very precious dye from the olden days. It became of universal significance when it was cultivated systematically on a large scale in the 17^{th} and 18^{th} centuries (Sandberg, 1989). The research on indigo dyeing is worth studying since it possesses a notable importance in the textile world.

To review, this research on the experimentation of indigo dyeing using different organic vats in indigo reduction is a study conducted to initiate environmentally safe approach to conventional indigo dyeing processes using chemical reducing agent. The study concerns about the reduction potential of each organic vat to produce good dyeing result and various indigo colour shades. The study also considers the influence of parameters of the organic vat dyeing process as to establish a well-developed research. The result of the research is important in producing apparel and furnishing products that come in different shades of blue. The outcome of the research could also be significant in the near future since there has been increasing interest in developing less polluted processes in textile industry.

The literature review will be reviewing the history of indigo and its chemistry as well as its reduction-oxidation mechanism. Conventional indigo dyeing, natural fermentation vat and previous research done to improve the environment unfavourable dyeing process will also be discussed in the next chapter to give a better understanding on indigo dyeing and plenty of ideas and knowledge to experiment with indigo dyeing processes.