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Comparative Analysis of Colour Strength and Fastness Properties on Extracts Natural Dye from Onion's Outer Shell and Its Use in Eco-friendly Dyeing of Silk Fabric

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Abstract: Silk fibre is one type protein fibre and its coloration is also an art. Every types of natural dyestuff can be used for silk dyeing. Acid dyestuff used for silk dyeing and required more than boiling temperature, resulting fibre damage and more energy. In this research, coloured silk fabric with natural onion dye for textiles by using a method of dyeing that relies on natural ingredients extracted from onion's outer shell colorant (Quercetin) by using water and organic solvents. And also used three types of mordants such as Alum, Copper Sulphate, and Potassium dichromate. The results of the research indicated that using onion's outer shell, deeper and more stable shades of the colours were obtained in comparison with water and organic solvent extracts dyed silk fabric. All samples were better results obtained such as light fastness, rubbing, water, acid and alkaline perspiration, changes appear in a smaller extent.

Keywords: Natural Dyes, Onion's Outer Shell, Mordants, Mordanting, Biodegradable, Silk Fabric

1. Introduction

Silk is a natural protein fibre of animal origin. "The dyeing mechanism of silk depends on free amino groups, carboxyl groups and phenolic with accessible -OH group [1]". Because of lightly cationic nature of silk with isoelectric point at P^H 4.5-5.5, it can be dyed with anionic dye such as all natural dyes, acid, reactive and selected direct dyes. But the main objective of coloration of a textile fibre is that the permanency of the colour and should not allow damage of natural abstract of fibre. "This implies that it should not destroy its colour during processing following coloration and dyeing & subsequent useful life (i.e. washing, light, rubbing, perspiration and saliva). Silk contain very small amount of sulphur [2]".

"For successful commercial use of natural dyes appropriate scientific techniques need to be established by scientific studies on dyeing methods, dyeing kinetics and compatibility of selective natural dyes in order to obtain shades with acceptable colour fastness behavior and reproducible colour yield [3]". "A renewed international interest has arisen in natural dyes due to increased awareness of the environmental and health hazards associated with the synthesis, processing and use of synthetic dyes [4]". Most of the natural dyes have no substantively for the fibre and are required to be used in conjunction with mordants. A mordant, usually a metallic salt, is regarded as a chemical, which will be fixed on the fibre and which will attach the dyestuff. "A link is formed in this way between the fibre and the dye [5]". Thus the use of natural dyes in textile application is growing and gaining popularity "because of their non-carcinogenic and non-hazardous nature [6], [7], [8]". "Extraction of colour component from natural sources is an important step for dyeing any textile substrate in order to evaluate their dyeing characteristics and maximize the colour yield on textile fabrics [9],[10]". Moreover, standardization of extraction process and optimizing the extraction variables both, "have technical and commercial importance on colour yield and cost of extraction process as well as dyeing cost [11]". As a result, "lead to extract the dye at low temperatures with minimum energy consumption and insignificant reactivity with pigments to avoid any loss in the colour quality [12]". "Onions can be used as natural dyes. Their scientific name is Allium cepa. Quercetin, commonly presented in the onion skin is the natural colorant that provides different brown tones to natural fabrics such as silk and wool [13]". Wool and silk fibres are protein-based and both have a general chemical formula NH_2.CHR.COOH. There are various functional groups that can be found in proteins, which determine the polymer properties. "The most important ones for the dyeing of wool and silk with acid dyes are COOH and NH_2 [14]". Protein is a sensitive fibre. Even dyeing with acid dyes at the boil caused some degradation, particularly on the prolonged boiling to promote dye migration. "This causes hydrolysis of various proteins [15]". "All natural dyes are eco-friendly and provide a wide range of attractive shades with acceptable level of colour fastness [16],[17],[18],[19]". In general, textile fibres can allow the adherence of the dyes in their structures as a result of vander waals forces, hydrogen bonds and hydrophobic interactions (physical adsorption). The uptake of the dye into the fibres depends on nature dye and chemical constituents. "The strongest dye-fibre attachment with an additional electrostatic interaction where the dye ion and fibre have opposite charges [20]".

1.1. Chemical Structure of Silk

Silk structure was shown in "figure 1 [21]".

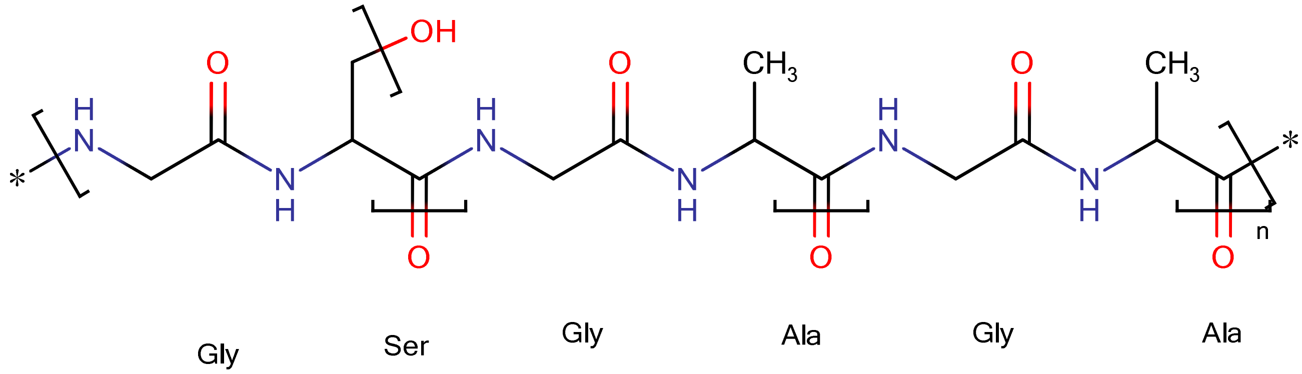


Figure 1. *Chemical structure of silk.*

1.2. Chemical Composition of Silk

Various types of chemical components are composed in the chemical structure of the fibre:

Table 1. *Chemical composition of silk fibre.*

Items	Amount
Fibroin	75%
Ash of silk fibroin	1%
Sericin	22%
Fat & wax	1.5%
Mineral salts	0.5%

2. Experimental

2.1. Materials & Instrument

Onion's outer shell, 100% (Silk fabric), Different types of chemicals, Milling machine, Automatic Heater (ZNHW HEATER Co.), Automatic Stirrer, Laboratory dyeing machine (MAT-24 M), Hot air dryer, Crockmeter (670 HD), Colour-fastness tester, light box (D65 artificial daylight), ISO grey scale for assessing change in shade (EN ISO 105-A03/IUF132/ VESLIC C 1211) and ISO grey scale for assessing staining (EN ISO-105-A05).

"Onion (Allium cepa) belongs to the Lilliaceae family and is grown all over the world. Yellow onion skins create a golden range of earthy colours. With a concentrated dye bath and enough time for the fibres to soak, the colours achieved are a combination of red and yellow, usually resting in the middle as an orange [22]". "The results radiate warmth and happiness, combining the physical energy and stimulation of red with the cheerfulness of yellow [23]". Onion outer skins are the most commonly discarded household and commercial food waste which can be used as dyes for colouring natural textile materials. These dyes, which are known as Quercetin (3,5,7,4'-tetrahydroxyanthocyanidin), work like acid dyes that can dye the protein fibres at high efficiency. The amount of Quercetin was found to be 2.25% in certain solvent extraction process using soxhlet apparatus. Due to presence of four hydroxy groups (Auxochrome groups) Quercetin exhibits good dyeing properties for dyeing of natural fibres. The molecular structure of onion skin dye (Quercetin) is shown in below figure [22]".

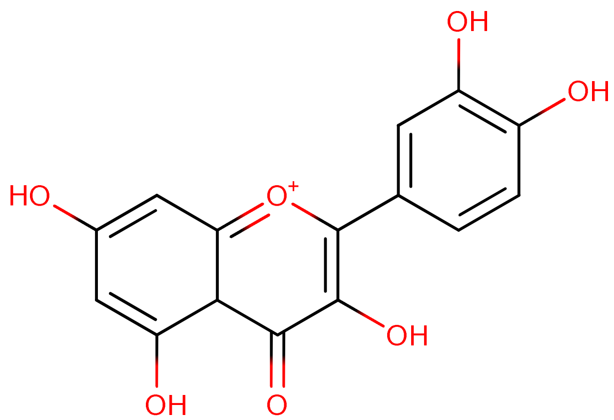


Figure 2. *Structure of Quercetin.*

2.2. Extraction of Dyes

As natural dye-bearing materials contain only a small percentage of colouring matter or dye along with a number of other plant and animal constituents such as water-insoluble fibres, carbohydrates, protein, chlorophyll, and tannins, among others, extraction is an essential step not only for preparing purified natural dyes but is also required to be carried out by users of crude dye-bearing materials. "As natural colouring materials are not a single chemical entity and the plant matrix also contains a variety of non- dye plant constituents, extraction of natural dyes is a complex process [22]". The nature and solubility characteristics of the colouring materials need to be ascertained before employing an extraction process. "The different methods for extraction of colouring materials are: Aqueous extraction, Alkali or acid extraction, Microwave and ultrasonic assisted extraction, Fermentation, Enzymatic extraction, Solvent extraction, Super critical fluid extraction [28]".

2.3. Degumming of Silk Fabric

Depending on the type, raw silk may contain 15-25% of sericin. "Although the sericin is responsible for silk's harshness, it is useful to manufacturer during the production process acting as a protective layer [24]". The removal of sericin occurs in a process called degumming, which can be entitled as a key process in which all sericin is removed, revealing softness and gloss of silk. In this research degumming was done by following recipe:

Table 2. *Degumming recipe for silk fabric.*

Dyeing recipe	Amount
Soda ash	3 gm/L
Standard soap	2 gm/L
Wetting agent	1 gm/L
Sequestering agent	1 gm/L
Material: liquor	1:20
Temperature	85°C
Time	40 minutes

2.4. Dyeing Recipe of Silk Fabric

Table 3. *Dyeing recipe for silk fabric*

Dyeing recipe	Amount
Onion dye (Ethanol / water extracted)	1%
Acetic Acid	1cc/L
P^H	4.0
Temperature	80°C
Time	60 minutes
M:L	1:30

2.5. Types of Mordant

a. Alum Mordant

Alum commonly called Aluminum sulphate is the most common mordant. It is classified as the brightening mordant, because it usually produces a pale and bright colour. It does not affect the colour being produced. Moreover, it can easily obtain from most chemists and is safe as well as cheap to use. Potash alum, which is the double sulphate of potassium and aluminum, is the most widely used aluminum mordant for natural dyeing. During dyeing, the aluminum then binds the molecules of the mildly acidic dyestuff, thereby creating the so-called lac which is insoluble. Hence, the dyes material is colourfast when washed.

b. Copper Mordant

Copper mordant is copper sulphate, sometimes it is called blue vitriol because it is available as a bright blue crystal. It is a one kind of dull mordants. Copper mordant also reacts with water to form a mildly basic hydroxide. Dyed textiles sometimes having a dull-khaki gold colour obtained from a plant will become a yellow-gold colour when treated with the alum, chrome and tin mordant respectively. However, the copper mordant produces a dull chestnut colour. It is mostly used for fixing the silk colours to produce brown shades. However, Copper mordant is less frequently used than the other mordants. In addition, it is more toxic. The disposal of small amounts of copper mordant and well diluted can reduce the environmental hazard.

c. Chrome Mordant

Chrome is known as potassium dichromate and is a kind of brightening mordant. It produces a deep version of the prevailing dye colour, and leaves silk with beautiful soft, while other mordants will harden the silk. Chrome is toxic and can cause irritation to skin. Care should be taken to avoid inhaling either the fumes of the mordant bath or the chemical dust. It is better to purchase chrome that looks slightly damp as opposed to a fine, as risk generated from the dust is greatly reduced.

2.6. Mordanting

Mordanting can be achieved by pre-mordanting (before dyeing), simultaneously mordanting and dyeing or post mordanting system (after dyeing). For mordanting the fabric at first we weigh the dry materials. Then rinse fabric with cold water. Then we took mordant, water and fabric in a vessel. For this project we used 5% Alum, 5% copper sulphate and 5% potassium dichromate.

1. Pre-mordanting Method

The textile material is first immersed into the mordant solution for 30 to 60 minutes at the room temperature to 100 °C with a liquor ratio of 1:10 to 1:20. The textile material treated with mordant is then dyed. After dyeing, the dyed material is washed with a non-ionic detergent. It is the most frequently used procedure by natural dyers because large quantities of textile can be treated and stored until dyeing.

2. Simultaneous Mordanting Method

As for the simultaneous mordanting and dyeing, the textile material is immersed in a dye bath solution containing both mordant and dye. Dyeing auxiliaries can be added during the dyeing process. For the optimization of dyeing condition, dyeing process variables can be studied for the specific fibre-mordant-natural dye system in order to improve the colour yield of textiles. After dyeing, the textile material is washed with the non-ionic detergent.

3. Post-Mordanting Method

The dyeing process is carried out on the bleached textiles in the dye bath without mordant. The dyed fabric then is treated with another bath called saturator containing the mordanting solution. Treatment condition may vary depending on the type of fibre, dye and mordant system being used. After dyeing, the textile material is washed properly with non-ionic detergent. When using this mordanting method, the colours are usually different and often less strong if the mordant and plant are boiled together. In all these three applications, mordants act as dye setters that will prevent the colour from running or streaking after dyeing.

2.7. Dyeing

Ethanol and water extracted dyeing with onion dye on silk used same method. The dyeing of samples carried out by using natural dye on lab dyeing machine keeping material to liquor ratio 1: 30. According to dyeing recipe firstly, water was added in the dye bath and then acetic acid was added to dye bath and pH was adjusted to 4.0 and 1% onion dye was added. Then degummed and pre-mordented silk fabric was entered in the dye bath. Then the temperature rose to 80°C in kept for 60 min dyeing. After 60 min dyeing completed then decreased temperature from 60°C to room temperature and dropped samples carried on post-mordanting.

It is necessary to follow each stage of the onion dyeing process with care. The stages are:

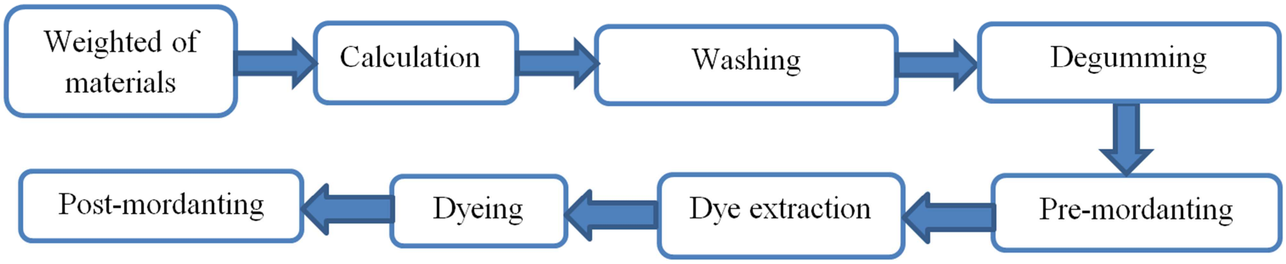


Figure 3. *Silk fabric dyeing process.*

2.8. Evaluation of Colour Strength and Colour Fastness

For "evaluation of the K/S value [25],[26],[27],[28]" of the undyed and dyed cotton and silk fabrics was determined by measuring surface reflectance of the samples using a computer aided Macbeth 2020 plus reflectance spectrophotometer, "using the following Kubelka Munk equation (1) [28],[29],[30]" with the help of relevant software:

$$K/S = \frac{(1-R\lambda max)^2}{2R\lambda max} = \alpha Cd \quad (1)$$

Where K = The coefficient of absorption; S = The coefficient of scattering; Cd = The concentration of the due and Rλmax = The surface reflectance value of the sample at a particular wavelength.

For "evaluation of wool dyed fabrics, the following tests were performed [25]".

- Colour fastness to washing, according to ISO 105-C01
- Colour fastness to rubbing, according to ISO 105-X12
- Colour fastness to light, according to ISO 105-B02
- Colour fastness to water, according to ISO 105-E04
- Colour fastness to Perspiration, according to ISO 105-E04

3. Result & Discussion

3.1. Optimization of Surface Colour Strength

The results obtained in Figure 3 indicate that colour strength values of silk fabric dyed with extracts of onion's outer shell colorant (Quercetin). And results show that dyeing performed at onion's outer shell sample gives maximum colour strength with darker shades.

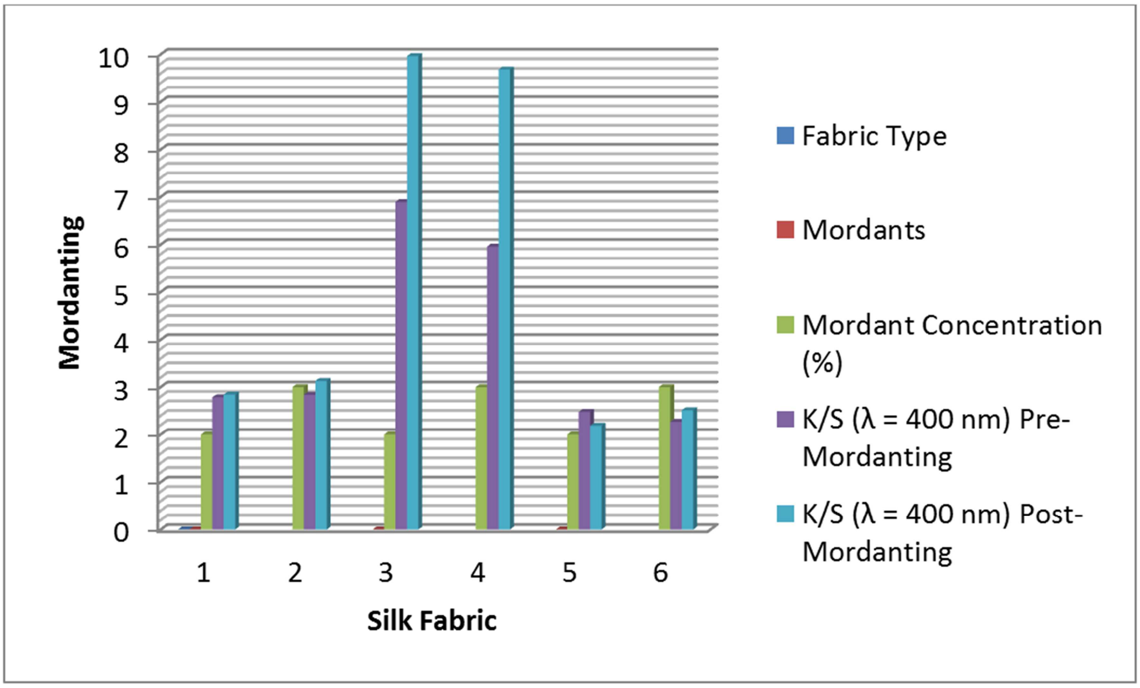


Figure 4. *Effect of pre-mordanting and post-mordanting on dying of Silk Fabric. Using extract obtained from onion's outer shell.*

3.2. Optimization of Colour Fastness

Table 4. *Colour fastness of Silk fabrics dyed using alum-mordant with methanol water and extracted onion dye.*

Fastness	Optimum conditions	Dyeing condition	Ethanol extracted dyed fabric	Water extracted dyed fabric
Washing	All silk fabrics dyeing temperature 80°C, Material to liquor ratio 1:30, Pre-mordant (3% Alum), Post-mordant (2%Alum)	Colour change	4-5	4
		Colour staining	4-5	4-5
Light fastness		-	4-5	4
Rubbing		Dry rub	4-5	4-5
		Wet rub	4	4
Perspiration (acidic)		Colour change	4-5	4-5
		Colour staining	4	4
Perspiration (alkaline)		Colour change	4-5	4
		Colour staining	3-4	3-4

In table 4 shows Ethanol and Water extracted dyed fabric using alum-mordant. Comparatively ethanol extracted dyed silk washing, light fastness, rubbing and perspiration (acid and alkali) were good.

Table 5. *Colour fastness of Silk fabrics dyed using copper sulphate-mordant with methanol water and extracted onion dye.*

Fastness	Optimum conditions	Dyeing condition	Methanol extracted dyed fabric	Water extracted dyed fabric
Washing	All silk fabrics dyeing temperature 80°C, Material to liquor ratio 1:30, Pre-mordant (3% copper sulphate), Post-mordant (2%copper sulphate	Colour change	4-5	4
		Colour staining	4-5	4-5
Light fastness		-	4-5	4-5
Rubbing		Dry rub	4-5	4
		Wet rub	4	4
Perspiration (acidic)		Color change	4-5	4-5
		Colour staining	4	4
Perspiration (alkaline)		Colour change	4-5	4
		Colour staining	3-4	3-4

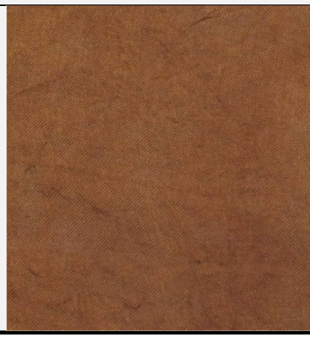
In table 5 shows Ethanol and Water extracted dyed fabric using copper sulphate-mordant. Comparatively ethanol extracted dyed silk washing, light fastness, rubbing and perspiration (acid and alkali) were good.

Table 6. *Colour fastness of Silk fabrics dyed using potassium dichromate-mordant with methanol water and extracted onion dye.*

Fastness	Optimum conditions	Dyeing condition	Methanol extracted dyed fabric	Water extracted dyed fabric
Washing	All silk fabrics dyeing temperature 80°C, Material to liquor ratio 1:30, Pre-mordant (3% potassium dichromate), Post-mordant (2% potassium dichromate)	Colour change	4-5	4
		Colour staining	4-5	4-5
Light fastness		-	4-5	4-5
Rubbing		Dry rub	4	4
		Wet rub	4	4
Perspiration (acidic)		Colour change	4-5	4-5
		Colour staining	4	4
Perspiration (alkaline)		Colour change	4-5	4-5
		Colour staining	3-4	3-4

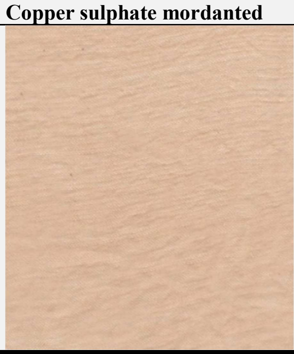
In table 6 shows Ethanol and Water extracted dyed fabric using potassium dichromate-mordant. Comparatively ethanol extracted dyed silk washing, light fastness, rubbing and perspiration (acid and alkali) were good.

Table 7. *Dyed silk fabrics (dyed with ethanol extracted onion dye).*

Fabric Type	Mordant type		
	Alum mordanted	Copper sulphate mordanted	Potassium dichromate mordanted
Silk			

In table 7 shows that potassium dichromate mordanted dyed silk deeper shad, copper sulphate mordanted and alum mordanted dyed silk medium shad.

Table 8. *Dyed silk fabrics (dyed with water extracted onion dye).*

Fabric Type	Mordant type		
	Alum mordanted	Copper sulphate mordanted	Potassium dichromate mordanted
Silk			

In table 8 shows that potassium dichromate mordanted dyed silk deeper shad, alum mordanted dyed silk medium shad and copper sulphate mordanted dyed silk light shad.

4. Conclusion

Use of water and organic solvents gave rise to onion's outer shell extract which are completely soluble. The best result of dyeing of the silk was achieved using a temperature of 80°C, pH 4.0 and 10 g/L initial dye concentration. To get good colour strength and darker shades, optimum dyeing conditions are 80°C, using material to liquor ratio of 1:30. Good colour fastness properties are obtained by employing alum 3% as pre-mordant and 2% as post-mordant onto silk fabric using extracts of onion's outer shell. It is inferred that rad-cure technology can also be successfully used for extraction of colorant from other dye yielding plants as well as application of synthetic dyes to get good colour strength and acceptable fastness properties of silk fabric. Tests of fastness to washing showed satisfactory results indicating that there was a good fixation of the dye in the fabric. Therefore, it was concluded that the silk fabric is easily dyed with extracts of onion's outer shell providing a yellowish brown colour. Considering that the dyeing process has a great contribution of chemisorption which promoted good colour fastness to laundering, it is concluded that dyeing with onion's outer shell has huge advantages as the textile wastewater in much more biodegradable than the textile wastewater generated with synthetic dyes. Finally, colour strength investigated the presence of mainly yield Quercetin (flavonoid) compounds in onion extract approach the demands of ecological viewpoints.

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Biography

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