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Green Synthesis of Dyes and Appliance on Silk by Using Metamordating Technique

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Abstract. The main idea of extracting dyes from plant (natural) sources is to avoid the environmental pollution. Natural dye from plants has been given much interest in recent years due to the threat and harmful effects are used by synthetic dyes and environmental awareness created by researchers. The drawback of synthetic dyes is that they are not durable as they do not completely fix on all kind of fabric and remaining dyestuff drained into rivers, becoming the cause of water pollution as well as they cause diseases in human being like cancer and allergies. On the other hand, natural dyes are eco-friendly and find use in the colouring of textiles, drugs and cosmetics *etc*. Owing to their non-toxic effects, they are also even used for colouring various food products. Mordents are used in natural dyes which enhance the colour of dye and stick it on fabric. Due to lack of availability of precise technical knowledge on the extracting and dyeing technique, it has not commercially succeeded like the synthetic dyes. Hence, the present study was planned to isolate eco-friendly dyes from the bark of the plants (*Ziziphus jujube* Mill., *Albizialebbeck* L., *Cordia dichotoma* G. Forst., *Ficus benghalensis* L. and *Cassia fistula* L.) and the roots of *Ficus benghalensis* L. Later on the dyes were applied on silk fabric by using metamordating technique. Then fastness properties like rubbing, heating, washing and sunlight were evaluated by using gray scale. *F. benghalensis* showed excellent fastness properties. *Z. jujube* showed series of colour variations on silk. Therefore, the bark and root of these plants can be successfully used for dyeing of silk. Natural dyes worldwide should be increased to prevent us from pollution and other harmful effects.

Keywords: natural dye, plants, eco-friendly, metamordant technique, silk fabric, bark, textile

Introduction

The art of dying is as old as our civilization (Kumar and Prabha, 2018). Before the discovery of synthetic dyes by William Henry Perkin in 1856, dyes obtained from plants were in use all over the world (Fan *et al*., 2018). Dyed textile remnants found during archaeological ex-cavations at different places all over the world provide evidence to the practice of dying in ancient civilizations. Natural dyes were used only for the colouring of textiles from ancient times till the nineteenth century (Kumar and Prabha, 2018). Afterward, synthetic dyes with low cost, plentiful colours, and easy access gradually replaced natural plant dyes. Nowadays, with increasing realization that some synthetic dyes can be harmful to health and the environment (Plácido *et al*., 2016; Shadeera, 2015; Mujadžiæ *et al*., 2014) there is renewed interest in natural dye-yielding plants (Shakeri *et al*., 2018). Furthermore, due to the severe threat of ecological globalization, environmental degradation and cultural homogenization, it is crucial to record the indigenous knowledge of plant utilization and preserve the plants' habitats, especially where they are not yet completely lost (Geng *et al*., 2016).

In the recent years synthetic dyes are totally being avoided to use because of environmental hazards (Bechtold *et al*., 2009; Samanta and Agarwal, 2009; Vankar *et al*., 2007). The European Union recently banned the use of azo dyes because of its harmful effects (Sivakuma *et al*., 2011). Many countries have eluded use of azo dyes in the textile industry. But the need of dyes in fabric industry is going to increase day by day so, there is a need to find out suitable and less toxic alternative sources of synthetic dyes (Paitoon *et al*., 2002). One such product from nature is the dye. Previously, pigment from leaves, fruits, seed, wood and roots were used as dye stuff for textiles and as paint in art and craft. Natural dyes are environmental friendly, hygienic, user friendly and permanent than other colourant. The replacement of natural dyes could happen until the introduction of synthetic dyes due to feasible colouring property of natural dyes (Kumaresan *et al*., 2011).

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Certain problems with the use of natural dyes in textile dyeing are colour yield, complexity of dying process, reproducibility results, limited shades, blending problems and inadequate fastness properties (Sachan and Kapoor, 2007; Siva, 2007). But these problems can be overcome by using chemicals called as mordants. Mordants are metal salts which produce an affinity between the fabric and the dye (Samanta and Agarwal, 2009; Vankar *et al*., 2009). One of the pleasures of using natural dye is that no baths will ever give exactly the same result, there will be an element of surprise with variation accordingly to the season, the weather, the maturity of the plant, its position in the sun or shade and the quality of the water used for dyeing will determine the dyeing quality (DivyaLekshmi and Ravi, 2013).

The dyers are trying to use natural dyes on regular basis both at small and larger scale to avoid environmental hazards caused by synthetic dyes. Natural dyes have no toxic effects. Therefore, it can be used in food products for colouration (Sivakuma *et al*., 2011). They have great tendency to bind with fabric. So, these dyes make a broad range of colours by using different plants part and apply on fabric like wool and cotton (Allen *et al*., 2008). They produce rare, peaceful and malleable colours unlike synthetic dyes (Allen and Bain, 2008).

Silk is composed of proteinous material which comes in contact with the body provide a suitable environment for the growth of pathogens and produce offensive smell and numerous diseases like dermal infections, product deterioration and allergies (Khan *et al*., 2011). As well as synthetic dyes used for dyeing silk are harmful for the mammalian cells and cause liver tumor. So, the demand of natural dyes is raised after knowing its therapeutic properties like it is antifungal, non-carcinogenic and antimicrobial (Chenghiah *et al*., 2011). Antimicrobial activity of *Turmeric terminalli* dye was checked and it was conclude that natural dyes were bound tightly with fabric and give good antimicrobial activity against fungal pathogens (Ranjit and Kalirajan, 2012).

Aqueous extract of *Hibiscus mutabilis* (cotton rose) flowers gave different shades of green colour with different mordents. These colours have been applied on silk fabric at commercial level. But the extraction from flower is difficult and only green colour range is produced. Mangrove bark is also used to form natural dyes because it has tannin. These dyes were applied on silk and gave only reddish-brown colour with different mordents (Nattaya *et al*., 2013). In the present work five plants species extracts were used to dye the silk fabrics at optimized conditions byusing a four mordant and checked the colour fastness of the dyed fabrics were also evaluated. The aim of this study was to isolate the natural dyes from the bark and root of plant species, to find the uses of different type of mordants and to check the effect of dyes on silk fabric by using meta mordating technique.

Material and Methods

For the extraction of dyes, green synthesis is preferable because this method is easy to perform, harmless and eco-friendly. The dyes used in this method are more reliable, consistent, give bright colour range and show different effect in the presence of different mordants. Following steps were used to perform this method:

Collection of plants. Fresh bark of the tree of *Ziziphus jujube* Mill., *Albizialebbeck* L., *Cordiadichotoma* G. Forst., *Ficus benghalensis* L. and *Cassia fistula* L. were collected from Pakistan Council of Scientific and Industrial Research (PCSIR) Laboratories Complex, Lahore, Pakistan, in November. Voucher specimens were submitted in Herbarium of Lahore College for Women University, Lahore, Pakistan.

Processing of plant material. All the selected part of the plants were weighed and washed to remove the dust particle. After sun drying material was used for extraction of dye.

Extraction of dye. The water was used as solvent for extraction. The plant material and water was mix in the ration of 1:100 w/v. Then, it was boiled on water bath approximately for 6-8 h until the final volume of aqueous extracts was reached upto 500 mL. The solution was filtered and stored at 4 °C in the refrigerator for further processing.

Dyeing of silk. Dyeing is the process to colour the textile fiber. The colouring substance should be the vital part of the fabric rather than an outer coating. The ratio of fabric and aqueous extract 1:20 w/v was used. Before dyeing silk fabric was dipped in water for 24 h. The required temperature for dyeing of silk was 100 °C for 1 h for complete dyeing.

Dyeing without mordant. For this purpose, 10 g piece of silk was dipped in the beaker containing 200 mL aqueous extract. Then boiled on water bath for 1 h at 100 °C with continuous stirring. Afterward the piece of silk was rinsed and colour was noted.

***Dyeing with mordants*.** A mordant is a substance, used to stick the dyes on fabric to produce different colours from the same extract. It can be added before and after or during process of dyeing. Metamordating technique was used for dyeing of silk. Different mordant like potash alum, copper sulphate, ferrous sulphate and potassium per magnate were used in the present study to develop different colours from a same extraction. Wet fabric (10 g) was dipped in 200 aqueous dyes and boiled on water bath at 100 °C for 45 min. Then 0.2 g mordant was added in and further boiled for 15 min. The fabric was thoroughly rinsed, washed, squeezed and dried at room temperature.

Verification of colour fastness properties. It was evaluated by following parameters:

***Washing*.** The transfer of colour from silk to wool and cotton was analyzed by using washing technique.

***Colour fastness to washing*.** Dyed silk fabric, wool and cotton were cut into 4×10 cm size pieces and sewed with each other in such a way that their faces were adjacent to each other. Took the 1:50 w/v ration of fabric and soap solution in the beaker then put on water bath at 80 °C for 30 min. Rinsed the fabric, checked the change in colour. Change in colour of specimen and staining of adjacent fabrics was assessed with the help of grey scale.

***Colour fastness to dry and wet rubbing*.** The colour fastness of silk against drying and wet rubbing was evaluated with help of Crockmeter instrument. Silk fabrics of 5 × 14 cm were fixed firmly by clamps to base board of Crockmeter. The piece of silk was first rubbed in dry condition and then in wet condition. Rubbing was done by to and fro motion in a straight line along a pathway of 10 cm long on exterior of fabrics 10 times in 10 sec with descending force on the finger rubbing. Fabrics were dried at 25 °C. Change in shade of the dyed fabrics and staining of the rubbing cloth was evaluated with the help of grey scale.

***Colour fastness to light*.** Weather meter instrument was used to evaluate the colour fastness to light. Weather meter is made by Atlas according to ISO 105 set method B02, in which the source of artificial light was Xenon arc lamp, representative of natural day light D65. Silk fabrics of size 7 × 12 cm were exposed to D65 light and result of colour fastness were determined with help of grey scale.

***Colour fastness to heat*.** Hand iron was used to check the colour fastness against dry heating on silk fabric. At specific temperature fabric was pressed for 5-10 min. Then change was noted by using grey scale.

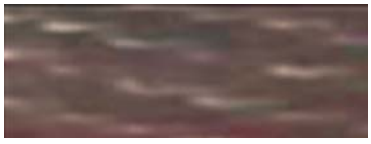
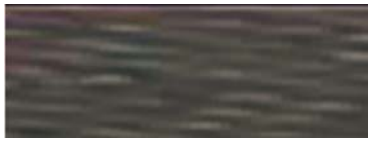
Results and Discussion

Bark of *Z. jujube*, *A. lebbeck*, *C. dichotomy*, *F. benghalensis* and *C. fistula* was used for silk dyeing. Root of *F. benghalensis* was also used for extraction of natural dye. Fixation of dye was done by four different mordants ($CuSO_4$, $KMnO_4$, $FeSO_4$ and Pot-Alum). Bark and root extraction with four different mordants resulted in production of 30 different shades. Fastness properties of all extracted dyes range between good and excellent (Fig. 1-5). The colour obtained from bark of *Z. jujube* extract was reddish. Five shades of this colour were produced with or without using different mordants. Pink with $CuSO_4$ dark blue with $FeSO_4$, dark brown with $KMnO_4$ and Pot were produce. Alum gave grayish brown shade (Table 1). The fastness properties like washing, rubbing, heating and light of these five shade ranged from 3.5-4 (Fig. 1). *A. lebbeck* extract was reddish brown. Different mordents gave different colours. Brown with $CuSO_4$, blue with $FeSO_4$, with $KMnO_4$ dark brown and Pot. Alum gave light brown colour (Table 1). Fastness properties like washing, rubbing, heating and light of these five shades ranged from 3-4 (Fig. 2). *C. dichotoma* extract was light brown. Different shades of brown were obtained with and without mordants (Table 1). Fastness properties like washing, rubbing, heating and light ranged from 3.5-4 (Fig. 3). The bark of *F. benghalensis* extract was red and with mordants gave bright colours while the root extract was brown in colour and with mordants gave dark shades (Table 1). The fastness properties like washing, rubbing, heating and light of *F. benghalensis* was in the range of 4-5 (Fig. 4). *C. fistula* extract was reddish brown and with mordants different shades of brown were obtained. The fastness properties like washing, rubbing, heating and light of *C. fistula*was in the range of 4-5 (Fig. 5).

Clothing is one of the basic needs of human beings. Silk fabric and other textile material contain moisture, nutrients and suitable temperature for the growth of microbes. So, there is a need to dye the fabric with material which prevents it from microbial attack (Singh *et al*., 2005). Natural dyes retard the growth of micro-organisms without producing toxicity (Han and Yang, 2005). Synthetic dyes are also useful to prevent the fabric from the microbial attack but synthetic dyes cause

Table 1. Silk colour variation of *Z. jujuba*, *A. lebbeck*, *C. dichotoma*, *F. benghalensis* (Bark and Root) in aqueous extract

Plant material	Solvent	Mordant	Silk colour
Ziziphus jujube (Bark)	Water	$CuSO_4$	
		$FeSO_4$	
		$KMnO_4$	
		Pot alum	
		No mordant	
A. lebbeck (Bark)	Water	$CuSO_4$	
		$FeSO_4$	
		$KMnO_4$	
		Pot alum	
		No mordant	
C. fistula (Bark)	Water	$CuSO_4$	

		$FeSO_4$	
		$KMnO_4$	
		Pot alum	
		No mordant	
C. dichotoma (Bark)	Water	$CuSO_4$	
		$FeSO_4$	
		$KMnO_4$	
		Pot alum	
		No mordant	
F. benghalensis (Bark)	Water	$CuSO_4$	
		$FeSO_4$	
		$KMnO_4$	

		Pot alum	
		No mordant	
F. benghalensis (Root)	Water	$CuSO_4$	
		$FeSO_4$	
		$KMnO_4$	
		Pot alum	
		No mordant	

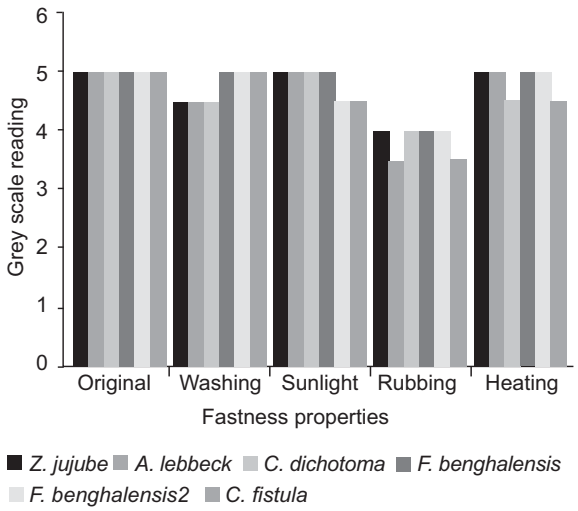


Fig. 1. Fastness properties of *Z. jujube, A. lebbeck, C. dichotomy, F. benghalensis, F. benghalensis(2)* and *C. fistula* without mordant.

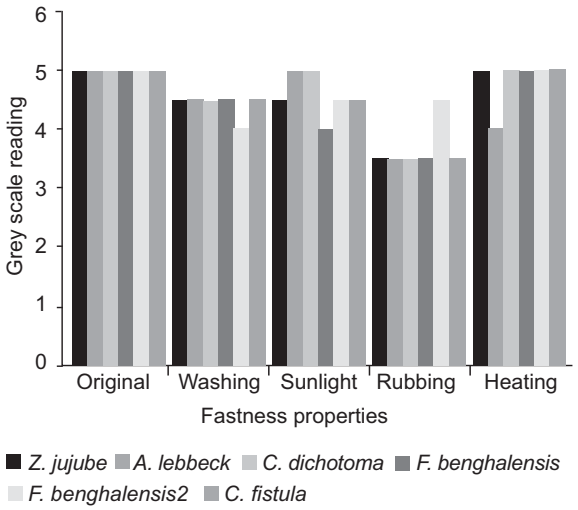


Fig. 2. Fastness properties of *Z. jujube, A. lebbeck, C. dichotomy, F. benghalensis, F. benghalensis(2)* and *C. fistula*with $CuSO_4$ mordant.

environmental pollution (Singh *et al*., 2005). In 1856 the first synthetic dyes were introduced. These dyes are widely used for dyeing of different types of clothes like leather, cotton, wool and silk fabrics but they are not eco-friendly and harmful for environment as well as for human beings. According to the United States EPA 5 to 35 gallons of water is required to dye fabric. Fibre absorbed the dye from solution this is called fixation rate. Fixation rate is not 100% so, remaining dyed solution is drained into river. The textile industry annually discharges 40,000 to 50,000 tons of dye into rivers and approximately 200,000 tons of salt which polluted the water.

To overcome these hazards plants extract are used for dyeing purpose because these dyes are eco-friendly, bio-degradable and harmless (Mongkholrattanasit *et al*., 2007). Many companies produced and supply natural dyes. De la Robbia in Milan produced natural dyes for textile industries under Eco-Tec system. Allegro natural dyes in USA also produced and supply natural dyes under E-colour label system (Hwang *et al*., 2008).

Characteristically, natural dyes have been reported manifold advantages. They have wide variety, non-carcinogenic, non-poisonous, bio-degradable and non-hazardous to life (Thiyagarajan *et al*., 2015; Saravanan *et al*., 2014). Unlike synthetic dyes, the natural dyes are renewable, non-toxic, sustainable and well known to create soft, subtle, pastel, soothing (Pervaiz *et al*., 2016; Kulkarni *et al*., 2011; Samantaa and Agarwal, 2009; Onal *et al*., 2005) and vibrant colours (Srivastava and Gautam, 2014). Natural dyes are neither harmful for human beings nor hazardous for environment (Shahid and Muhammad, 2013). Plant based dyes are reported economical (Pervaiz *et al*., 2016 a, b and c; Jadhao and Rathod, 2013), and easy to handle, render fragrance in the dyed substrate (Pervaiz *et al*., 2016 a, b and c),

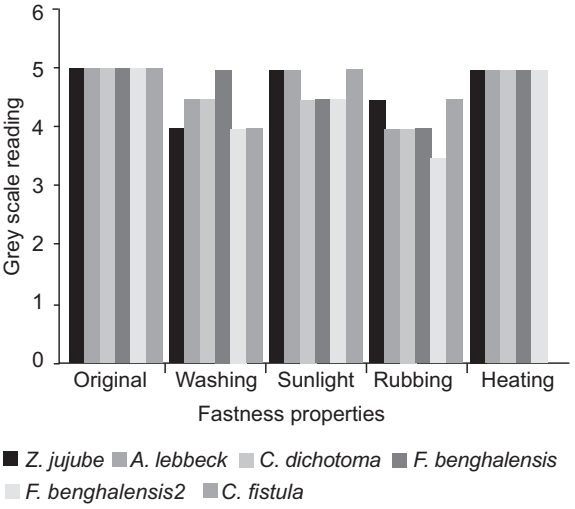


Fig. 4. Fastness properties of *Z. jujube, A. lebbeck, C. dichotomy, F. benghalensis, F. benghalensis(2)* and *C. fistula* with $KMnO_4$ mordant.

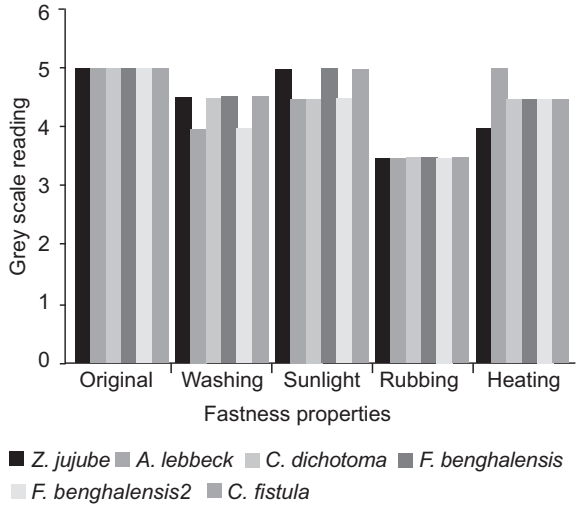


Fig. 3. Fastness properties of *Z. jujube, A. lebbeck, C. dichotomy, F. benghalensis, F. benghalensis(2)* and *C. fistula* with $FeSO_4$ mordant.

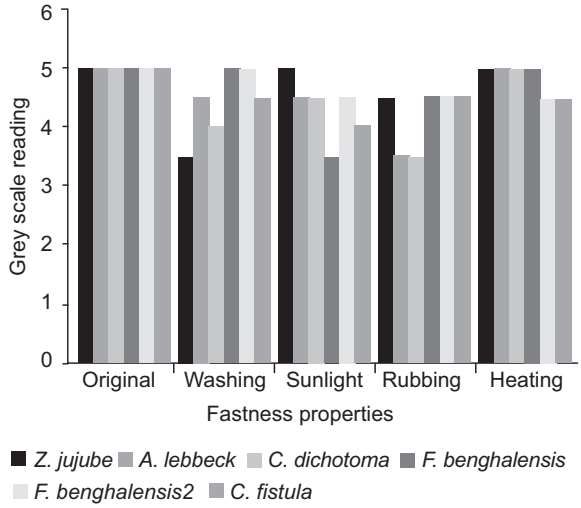


Fig.5. Fastness properties of *Z.jujube, A. lebbeck, C. dichotomy, F. benghalensis, F. benghalensis(2)* and *C. fistula* with potash alum mordant.

helpful to generate employment, safe for ecology (Mahanta and Tiwari, 2005), provide economic benefits through sustainable yield and harvest of dye bearing plants (Saravanan *et al*., 2014). On the other side, commercialization of natural dye can help to boost up the economy of the country (Belemkar and Ramachandran, 2015; Saravanan *et al*., 2014; Upadhyay and Choudhary, 2014; Ghorpade *et al*., 2000) which has an opportunity to earn carbon credits by reducing consumption of petroleum based synthetic dyes (Aishwarya, 2014).

In present study plant bark is used for the extraction of dye which is considered as wasted part of plant and is easily available. The use of bark did not lead the threat or extinction of plant, while in other study *H. mutabilis* flower used for extracting natural dyes and applied on silk fabric (Rakhi and Padma, 2005). Flower is an important part of plant and first source for the production of new plant. They used cotton rose for extraction of dye which is medicinally important and use for treatment of skin (Dasuki, 2001). Moreover, large number of flowers is required for colouring the fabric which may lead the extinction of plant. Natural dyes are preferred over synthetic dyes because of its harmless nature. So, the selection of mordants is much important in the present study $CuSO_4$, $KMnO_4$, $FeSO_4$ and Pot-Alum used as mordant. These mordants are non-toxic and easily degradable, while in other study *F. religiosa* L. bark was used for the extraction of dye and applied on silk fabric (Saravanan and Chandramohan, 2011) but they used stannous chloride as mordant which is responsible for human DNA damaging because it quickly receive by white blood cells of human being (Mclean *et al*., 1983).

After dyeing the evaluation of fastness properties (washing, heating, rubbing and sunlight) was also very important part of the this study. These properties were checked by gray scale. All the fabric with or without mordant showed the properties between good to excellent while in other study mangrove bark is used to form natural dyes and applied on silk fabric (Nattaya *et al*., 2013). They used stannous chloride as mordant which showed very poor light fastness property on silk fabric and ferrous sulphate showed poor washing property (Pervaiz *et al*., 2016 a, b and c).

Conclusion

This study concluded that natural dyes extracted from bark of *Z. jujube, A. lebbeck, C. dichotomy, C. fistula, F. benghalensis* and the root of *F. benghalensis* can be successfully applied on silk in the presence of four mordants ($CuSO_4$, $KMnO_4$, $FeSO_4$ and Pot- Alum). Almost 30 different shades can be produced from the same extract. The mordant treated dyes have good colours and wash fastness. The strength properties of all extracted dyes range between 3 to 5, shown by *C. fistula*. The highest fastness property which range is in 4 to 5. It can also be concluded that natural dyes are found environment friendly, low cost, non-carcinogenic, non-hazardous and very useful for silk dyeing. The results of silk dyeing with natural dyes by using different mordants were found encouraging in terms of their colour fastness to light, heat, rubbing and washing properties. Therefore, there is a good scope for Pakistan's silk industry to use natural dyes to meet the fundamental requirement of European Union, to earn carbon credits by reducing the use of synthetic dyes and to improve the socio-economic condition of farmers by yielding and harvesting dye bearing plants. Therefore, the role of the government should be to encourage the use of natural dyes by providing soft loans and financial support to farmers for the cultivation of dye bearing plants. In addition, government can provide assistance in marketing of natural dyes and reward the environment friendly silk industries.

Conflict of Interest. The authors declare no conflict of interest.

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