Chapter I Introduction

1. Introduction

1.1. What is silk?

 Silk is a type of fabric of natural origin. Silk's incorporation into the textile industry can be attributed to its natural luster, pliability, eco-friendliness, and mechanical strength [1-3]. The structure of silk closely resembles that of human skin; its smooth, semi-permeable, soft, non-pinching, etc. properties make it an ideal resource for excellent clothing [4-6]. Silks are protein polymers that Lepidoptera order larvae, such as silkworms, spiders, mites, etc., spin [7]. Silks are made in the epithelial cells of the silk gland, which is similar to the salivary gland of Drosophila in terms of evolution [8]. The proteins are then sent to the lumen of special glands, where they are stored before being spun into fibers [8]. The source of silk plays a crucial role in the determination of the composition, structure, and properties of the silk. Some of the known types of silk are as follows:

- 1. Pat silk (Bombyx mori): It is known to be one of the most common silks famous for its fine texture and durability. The silkworms producing these silk feds on mulberry leaves [9].
- 2. Tussar silk (Antheraea mylitta and Antheraea Proylei): It is a wild silk produced by wild silkworms. It has a natural golden sheen with a coarser texture. It is also one of the non-mulberry silks.
- 3. Eri silk (Samia ricini): Silk produced by Eri silkworm that feeds on the castor plant is called Eri Silk. They are also termed ahimsa silk. It is mainly worn during winter [10, 11].
- 4. *Muga silk (Antheraea assamensis)*: It is known for its golden shine. The silk is exclusively found in Assam [12-14].

India produces all the major commercial varieties of silk [15]. Each of the silks has its signature that makes them different in terms of purpose, from attire to textile applications [15]. Silk is also classified based on geographical distribution, and some notable silkproducing regions and the associated silk types associated with them are:

- 1. China: It produces different types of silk that include Pat, Tussar, and silks from other types of silkworms. It is the world's leading producer of silk.
- 2. India: India is another major silk-producing country. Pat silk, Tussar silk, Muga

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silk, and Eri silk are among the famous silks produced here. The specification of silk is again distributed among different regions in India [16].

- 3. Italy: Como and Lombardy in Italy are famous for their high-quality silk. It has its demand in high-end fashion and textiles [17].
- 4. Thailand: It is known for producing Thai silk from native silkworm species. It is widely famous for its vibrant colors and intricate patterns [18].
- 5. Brazil: A unique type of silk is found in Brazil which is called Brazilian silk or wild silk. It is characterized by its natural color variations and texture [19].

Apart from these examples, there are other countries as well that are known for silk production. The specificity of silk varies depending on the local climate, availability of silkworms, and historical traditions of each region [20].

Based on purity, silk can be pure, monohybrid, or poly-hybrid, which refers to the cross between two or more of its strains [3, 21]. However, purity is mainly based on mixing, which divides it into pure silk or blended silk. Pure silk refers to silks that are entirely made from the cocoons of a specific species of silkworm [22]. The fine texture, natural luster, and durability make it a valued possession. It is specified to be "pure silk" or "100% silk". On the other hand, blended silk (mixed silk) refers mainly to silk that is combined with other fibers. The blending leads to specific characteristics, which also contribute to lower costs. Commonly found blends include silk-cotton, silk-polyester, or silk-rayon [23]. Here, the properties of silk are being enhanced, like strength, drape, and affordability. The product description generally indicates the silk blend on the label for such blended silks [23-26].

Silk is said to be one of the most well-known textile filaments. Silk is referred to as the "queen of textiles' because of its remarkable properties, which include:

- 1) Strength: Silk has a high strength that increases its demand.
- 2) Luster: The natural luster of silk can't be matched to other fabrics.
- 3) Dyeability: A wide range of colors can be used to dye silk.
- 4) Comfort: It is very soft and comfortable to wear [27-29]

 It is in demand for style apparel. As it is a protein fiber, it is considered the only biologically origin fiber found in filament shape [30]. The instinctive properties of silk are strong, extensible, and mechanically compressible [31]. The rearing of silkworms

for producing its cocoons, which are the basic material in producing silk is termed sericulture [32].

1.2 Composition of Silk

The diet of silkworms must contain four main constituents: fiber, saccharides, water for the nourishment of the worm, and resin for preparing silk. The spinning organ is located along the side of the alimentary canal. The glands have 3 parts:

a) anterior (length: 2cm, secretory cells: 250) that produces sericin,

b) middle (length: 7cm, secretory cells: 300) that secretes sericin, and

c) posterior (length: 15cm, secretory cells: 500) that secretes fibroin [33].

The lumen of the silk gland stores the liquid silk synthesized in the glands. The fluid content of the glands comes out and gets transformed into a thread in the presence of air, called silk [33, 34]. The fully grown larvae stop eating and search for an optimal place to begin spinning a cocoon [33]. For spinning, they hold their body tightly by ten hind legs and move their frontal body [35]. The silk proteins that silkworms produce are fibroin and sericin [34]. The structure of silk filament adapted from Sonthisombat and Speakman is shown in Fig 1.1. Fibroin consists of 72–81% of the cocoon, and the remaining 19– 28% is sericin [33, 36]. Additionally, it contains:

a) 0.8–1.0% of fat and wax;

b) 1.0–1.4% of colour materials;

c) ash

During spinning, two exocrine silk glands make twin strands of fibroin. At the same time, the strands are stuck together with sericin, which gets stronger and harder when exposed to air [37]. The amino acid makeup of the fibroins of *Eri*, *Muga*, & *Pat* discussed in the next section [37, 38] is given in Table 1.1 [39].

Amino acid	Samia ricini	Antheraea	Bombyx mori
	(Eri) (mol %)	assamensis	(Pat) (mol%)
		$(Muga)$ (mol%)	
Aspartic acid	3.89	4.97	1.64
Glutamic acid	1.31	1.36	1.77
Serine	8.89	6.11	10.38
Glycine	29.35	28.41	43.45
Histidine	0.75	0.72	0.13
Arginine	4.12	4.72	1.13
Threonine	0.18	0.21	0.92
Alanine	36.33	34.72	27.56
Proline	2.07	2.18	0.79
Tyrosine	5.84	5.12	5.58
Valine	1.32	1.5	2.37
Methionine	0.34	0.32	0.19
Cystine	0.11	0.12	0.13
Isoleucine	0.45	0.51	0.75
Leucine	0.69	0.71	0.73
Phenylalanine	0.23	0.28	0.14
Tryptophan	1.68	2.18	0.73
Lysine	0.23	0.24	0.23

Table 1.1: Amino acid composition of Eri, Muga, and Pat [39]

Fig 1.1 : Structure of silk filament [40]

Silks are genetically displaying a mixing of frequent repeats of primary sequences that contribute to the secondary structure's significance and show unique mechanical properties. Silks' biocompatibility and other properties led to their use in guided delivery and other applications [41, 42].

The molecular formula for silk fibroin can be written as (-CHR-CO-NH-) n, where $n = 1100$. Here, R is any of the many amino acid residues found in fibroin's building blocks. The polypeptide chain contains both a backbone and side chains composed of amino acid radicals R. Depending on the amino acids present in the protein, the level of branching of the chain varies. The hydroxyl groups of serine residues, on the other hand, are less accessible to chemical reagents. Consequently, the side chains constitute 19 percent of the fibroin weight. For instance, hydrocarbon residues have side chains that are not polar [37] and may include polar groups like the ones in [37].

1.3 Assam Silks

The state of Assam $(78,438 \text{ Km}^2)$ is a part of North-East India with a latitude of $24^{\circ}8'$ N to $28^{\circ}2'$ N and a longitude lying between $89^{\circ}42'$ E to 96° E, which has a rise of 45-1964 m from the sea-level [43]. Assam is well known for its tropical monsoon rainforest with high levels of rain and humidity [44]. An adequate environmental moisture level is required for healthy cocoon formation and silk production, which is met in Assam. The temperature of Assam revolves around 8° C to 20° C, whereas summers lie between

32°C and 38°C. Annual rainfall ranges from 1500 mm to 3750 mm, witnessing heavy rain resulting from the southwest monsoon, mainly from May to September [44]. Apart from the weather, the divergent vegetation of Assam plays a crucial role in providing the primary food source to different varieties of silkworms. The geographical location and its weather best serve the growth of silkworms.

Silks that are dominating in Assam are Eri, Muga, and Pat [45]. Eri and Muga silks vary in luster, color, and tensile properties as the silkworms are found in polymorphic forms growing various host plants distributed in different geographical regions. These silks are related to the Saturniidae family [46]. The golden yellow silk, Muga, is produced by the silkworm Antheraea assamensis [47]. The color of Muga silk is related to the plant it feeds on and the season. The name $Muga$ means yellowish in the language of Assam [47]. The species originates in the north-eastern part of India. Eri, produced by Samia ricini, is widely used in making traditional household clothes in the northeastern states of India [45]. Pat is related to the Bombycoidea silkworm family. Bombyx mori is the single species used to produce commercially available pat [45, 48]. The classification of the three silks has been presented in Fig.1.2.

The practice of silk production is termed "sericulture," which holds significant importance in Assam's economy by providing employment opportunities and also contributing to the socio-economic well-being of the region. Sericulture in Assam mainly comprises Eri, Muga, and Pat. Eri and Muga are considered to be indigenous to Assam [45]. *Eri* fabric is coarse, fine, and dense. It is heavier than *Muga* and *Pat. Eri* is cultivated mainly in Assam and in the seven states of northeast India. Eri has insulating properties, making it special for winters too. Assam contributes to 65% of *Eri* production in India [37, 49]. Muga fabric is glossy with a fine texture and durability [50]. Since 2007, Muga silk has had the Geographical Indication (GI) status, and the logo for genuine manufacture registered with the Assam Science, Technology, and Environment Council [51]. Assam contributes 95% of its production to India [47]. Pat silk is light, delicate yet strong, and also resilient. The silk has a shiny white shade and is known for its longevity and glossiness. It is primarily used for manufacturing dresses [52]. It is produced mainly in Karnataka, Andhra Pradesh, West Bengal, Tamil Nadu, and Jammu and Kashmir, making 92% of the country's total production[53]. Assam and a few other states together contribute to the rest of the production of Pat (mulberry) silk in India. The climate, vegetation, and cultural heritage together contribute to the thriving sericulture industry in Assam. Various organizations and institutions collaborate with the state government to promote sericulture in the state through training, research, and development programs.

Eri silkworm (S. ricini) eats the leaves of the castor plant (Ricinus communis), also known as Era , thus getting its name Eri [45]. However, they are known to have voracious appetites and are polyphagous (feeding on a variety of host plants), including sources like castor leaves, tapioca leaves (*Manihot esculenta*), and some other leafy plants [54]. Muga generally feeds on Som (Persea bombycina) and Soalu (Litsea monopetela). Secondary host plants of Muga are Mejangkari (Litsea cubeba), Pan Chaapa (Magnolia sphenocarpa), and Digholati (Litsea salicifolia). Mulberry silkworms have excellent adaptability to mulberry *(Morus alba)* tree cultivation, which they exclusively feed on.

Eri silk, which is known for its soft and warm properties, has a texture similar to wool. Due to its insulating properties, it is suitable for winter garments. The silkworm falls into the family Saturniidae (giant silk moths). *Eri* silks are known to have a distinctive feel and drape and often exhibit a slight natural sheen. Eri, produced by S. ricini, is widely used in making traditional household clothes in the northeastern states of India [45, 55, 56].

The golden yellow silk *Muga* is produced by A. *assamensis*. This silk is used in crafting traditional garments like *mekhela sador*, worn by women, and jackets worn by men during special occasions and festivals. Muga silk is also associated with royalty, elegance, and heritage [57].

Pat silk is known for its fine and smooth texture. It is not just considered the most luxurious but also a prized silk variety. It is mainly used for high-quality textiles. Pat is related to the Bombycoidea silkworm family. They require the intervention of humans for their maintenance, so they are entirely domesticated [45, 48]. The fabric and threads of *Eri*, *Muga*, and *Pat* are shown in Fig.1.3.

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Fig 1.2: Classification of Eri, Muga, and Pat

Fig 1.3: Image of untreated thread and fabric of a) Eri; b) Muga; and c) Pat

1.4 Silk processing

Various chemical and physical alterations are made to cocoon-stage silk to create garments. The processing begins with cocoon cooking or boiling, where the cocoons are swelled and softened, and sericin is dissolved a little. Following this is a collection of fiber ends that are reeled together by adding a new end to the previous end to maintain the yarn's continuity. This reeling includes deflossing, end-pulling, joining, rotating, reeling, and re-reeling [37, 45]. For example, in the case of Muga silk, the cocoon for

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reeling is mainly collected from the "Kotia" (autumn) crop, as during this phase, the leaves are suitable, and the cocoons are rich in silk along with low seasonal wasps and flies [58]. These are then subjected to weaving to prepare the silk cloth. Different chemicals and methods are used for different silks. Degumming (deserialization) is a simmering process during which the silk sericin is scoured from the fibroin. In degumming, the sericin is scoured from the fibroin using an alkali and/or acidic solution, after which the fibers become luxurious with excellent drape, wonderful luster, and a glossy facet, making them highly demandable in the textile field [31, 59]. Degumming causes 20–25% of the weight loss of the silk threads [37, 59-61]. Commercially, many agents are being used, such as the soap-soda ash method, tartaric acid, oxalic acid, citric acid, etc. [61-64]. Traditional agents that are being used with comparatively effective results are *Citrus limon* (lemon), *Dillenia indica* (elephant apple), and *Musa balbisiana* (banana) [59, 63-68]. The reeling process is different for the three silks. The silk weaving follows it. There are different looms and accessories, which are done both commercially in factories and also traditionally at the houses of the weavers. The throw-shuttle loom (country loom) and the loin loom constitute the traditional looms of Assam. Apart from these, small appliances called *Letai, Chereki,* and *Ugha* made of bamboo are also used traditionally in Assam in the process of manufacturing fabric. The fly-shuttle loom is an improvised throw-shuttle loom mainly used in factories that are accessorized with drawboy, dobby, and jacquard machines to create elaborate designs [45, 69].

Differences in the preparation methods for the three silks can be compared. For cooking, Muga is boiled in an alkaline solution prepared from plantain ash and straw ash [45, 70]. Eri cocoons are boiled in a 10% sodium solution; in some places, ash obtained from banana leaves, wheat stalks, paddy straw, and pieces of green papaya are commonly used [71]. In some cases, Eri cocoons aren't cooked but just brushed manually to get the ends. 'Kolakhar, made from Musa balbisiana, is reported to contain potassium carbonate in large quantities [72]. Citrus limon and Dillenia indica, on the other hand, contain citric acid as the main compound. These are mainly used for degumming *Muga* silk [59]. For *Eri* degumming, sodium carbonate and neutral soap are commonly used [71].

Earlier, after degumming, Muga reeling is carried out in "Bhir", however in recent years, the use of pedal-driven reeling machines (RMRS type, Choudhury type),

'Bani' reeling machine, and motor-driven machines (CSTRI reeling cum twisting machine) are also used [58]. Re-reeling the raw silk results in the production of yarn in hank forms, whereas in *Eri* spinning, which uses the *Takli* method and a spindle with a disc-like base, produces yarn in spindle form [73, 74]. For *Pat*, the processes are quite different. The cooking of the cocoons is generally done in plain water at 90^0C-95^0C for about 15 minutes. Here, reeling is done without the addition of any chemicals in distilled water at 40^0C-45^0C . The process of degumming includes a sodium carbonate and sodium silicate mixture with any non-ionic wetting agent at around 90° C for 3 hours [75, 76].

1.5 Popularity of handloom weaving in Assam

The fame in the textiles market at domestic and international levels has been mentioned in several eras of Indian history. As per the annual report of 2016-17 of the Ministry of Textiles, Government of India, the Indian handloom has an important position in the export market, sharing 95% of the world and being exported to 125 countries. For handloom products, India is the second largest exporter in the world as per 2017-18 reports [77]. In the entire country, more than 38 lakh handlooms are present; out of these, 15 lakhs are present in northeast India. Interestingly, out of the 20 major clusters of handlooms in India (with handlooms >50,000), Assam holds 14 of them. Handloom weaving is an age-old cultural component of Assam. The weaving tradition includes both self-consumption and commercial purposes. Assam is the only state in India that produces all four commercial types of silk (Eri, Muga, Pat, and Tasar). As per the press release by the Ministry of Textiles (December 2018), in the year 2017-18, 4645 MT Eri, 157 MT Muga, and 59 MT Pat silk were produced in Assam. Assam has 13 Muga seed farms that consist of 236.21 ha. Government Eri seed grainage in Assam is 26 in number and is known to provide disease-free seeds, which accounts for 194.29 ha. There are 109 collective mulberry (Pat) gardens (CMG) that consist of 697.02 ha [78].

Imparting color on silk fibers, fabrics, or garments is called dying of silk [79]. Before dying, silk needs to undergo pre-treatments to remove impurities or finishes that might interfere with dye absorption, which includes degumming or bleaching, depending on the requirement [80]. Both natural dyes (indigo, turmeric, or cochineal) and synthetic dyes that are specifically formulated for silk are being used [81]. The choice of dye is an important factor as it is associated with desired colors, fastness

properties, and dyeing techniques. In these, techniques like immersion dyeing, direct applications or printing, and resist dyeing techniques like tie-dyeing are being used [82, 83]. The dye is applied through many processes and is subjected to appropriate heat or chemical treatments to fix the dye onto the surface and enhance colorfastness. Additional processes are being applied to improve colorfastness and remove any residual dye or chemicals. Apart from this, the complete textile dyeing industry creates pollution issues being a chemically intensive industry on earth and a major source of pollution of clean water. For bleaching, chlorine bleach is too toxic to the users and its surroundings, yet it is still used for bleaching fabrics. An alternative and less toxic bleach should always be encouraged [84, 85]. To avoid damaging fabric, the dyeing process needs to be carefully controlled.

1.6 Post-Production Limitations

The silk fabric produced faces a lot of hardship for its maintenance. Silk fabrics crease, leading to dimensional deformation when subjected to some home laundering. During the rubbing method of laundering, fibrillation and degradation occur, which fracture the fiber. This fracture affects the appearance and wears endurance of silk garments and leads to a loss of quality. Durable press finishing for creases leads to fabric style changes and hand property losses [86-88]. Dry cleaning silk is costly and relatively inefficient at removing stains. The knitted fabric undergoes dimensional change as yarns relax and loops reorient during the manufacturing process [89]. After 10 cycles of washing, silk yarns may develop hairiness, which may suggest damage to the silk fibril as a result of mechanical action during laundering [90]. According to research, several variables, including fiber characteristics, stitch stretch, machine gauge, yarn joints, knitting tensions, and wash-and-dry techniques, influence the shrinkage of knitted fabrics [91, 92]. Thus, the post-production limitations of silk can be pointed out as follows [93]:

- 1. Delicate nature: This property leads to easy damage by rough handling, repeated friction, or using of harsh chemicals. Hence, special care is required during washing, drying, and storing silk apparel.
- 2. Heat sensitive: Silk is known to have less heat resistance and gets easily damaged at higher temperatures, which makes it unsuitable for ironing as it can lead to scorching or weakening of the fibers.
- 3. Removal of stain or spot: Cleaning of spots or stains may lead to colour differences and even permanent damage.
- 4. Fading: When exposed to direct sunlight, silk may experience fading or color change over time.
- 5. Cost and availability: To produce silk, significant resources and labor are required. Specific geographical requirements and limited availability of silk-producing silkworms contribute to the relatively higher price of silk in comparison to other fabrics.

The limitations mentioned above can lead to the following issues [88, 93-95]:

- 1. Silk Shrinkage: Due to protein denature when washed or dry-cleaned, silk is prone to shrinkage.
- 2. Silk Wrinkling: Due to its smoothness that lacks friction, silk is associated with wrinkling.
- 3. Color bleeding: Natural fibers can release their colour when exposed to water or heat.
- 4. Pilling: Over time, silk tends to pill, which is due to its soft fiber that can catch on other fabrics.

Despite these limitations, silk remains highly desirable for its luxurious feel, natural luster, and breathability. With proper care and attention, silk products can maintain their beauty and longevity.

1.7 Biodegradation of Silk

Despite the environmental problems brought on by the disposal of nonbiodegradable textiles, their manufacturing is rising. With the rise in textile waste, landfill sites are becoming a bigger concern for environmental preservation. Degradability has gained attention as the recycling of non-biodegradable textiles has been discouraged. It's crucial to consider how naturally fabrics degrade in soil [96]. Numerous microorganisms, including fungi, bacteria, worms, and many species, assault the materials during biodegradation. It is possible to observe both good and negative impacts of the biodegradation process, such as the removal of wastes, the provision of nutrients for new life, the generation of energy required for various biological processes, as well as the potential for environmentally hazardous waste [96-100]. Through visual inspection, measurement of mass loss, characterization of the chemical composition, and surface appearance, biodegradation was specifically assessed. Biodegradable materials are the preferred choices for the development of therapies. Due to its unique crystalline orientation and structure, silk fibroin has a tight structure and is challenging to break down. Proteolytic enzymes more efficiently digest silk fibroins with low molecular weight and non-compact structures than other enzymes do. Enzymes have an impact on the biodegradation of silk fibroins. According to reports, chymotrypsin and protease cocktails can enzymatically break down silk [101-103].

1.8 Hemocompatibility of silk

 When developing biomaterials that come into direct contact with blood, hemocompatibility testing is crucial and frequently performed. It is important to increase hemocompatibility by topological change or re-modeling the blood-contacting materials or implantable devices [104]. Plasma proteins compete with one another for space on the surface of a foreign biomaterial when blood comes into contact with it. Numerous studies have focused on surface modification as a means of enhancing biomaterials' hemocompatibility [105]. To reduce protein adsorption and, ultimately platelet adhesion, hemocompatibility on a biomaterial surface is improved. The main cause of hemolysis is an increase in the osmotic pressure of red blood cells (RBCs). Hemolysis causes the rupture of RBCs and the release of hemoglobin from RBCs. The percentage of hemolysis (HP) indicates how many red blood cells (RBCs) were broken by the sample when it came into contact with whole blood [106]. The value of the hemolytic ratio increases with the number of broken red cells [105, 106].

1.9 Gap in the study

 The study on proper silk washing is limited; however, some studies that were carried out indicate fibrillation and degradation of silk during the laundering process, which affect the appearance and wear endurance. Users have struggled with safe silk washing for a very long time. The demand to develop methods for washing such delicate fabrics needs to be studied.

It's a major challenge for the textile industry to produce long-lasting, vivid colors as the color fastness of washed dyed silk fabric is relatively low. There has been some success in stabilizing colors in silk by using metal salts as mordants, but this leaves mordants in the wastewater, which poses toxicological and cancer-causing risks. The successful use of natural agents in the entire dyeing process of Assam silk is scanty.

There have been studies on the proteolytic degradation of silk, but the soil burial method to examine the biodegradation of Assam silk is lacking. Silk as a biomaterial has been in use for many decades; however, it is limited to Pat silk. Many national regulatory frameworks mandate performing a biological safety assessment before moving from device assessments to first-in-man clinical trials. Thus, the study of biocompatibility is the preliminary step for such studies. Hemocompatibility testing is common and essential during the development of biomaterials that will come into direct contact with blood. In addition, biomaterials derived from silk fibroin have been utilized in biomedical applications for over a century, and their use is accelerating. To open the door for Assam silk to be used as a biomaterial, more research on its biocompatibility is required.

1.10 Aims and objectives of the present study:

Taking the mentioned problems into account, the effects of laundering on silk fabrics of some commonly available detergents, conditioners, stiffeners, traditionally used ingredients, etc., were compared. The physicochemical changes were reported to compare the effects of the materials used, which suggests a comparatively better option for silk washing. The entire dyeing process of bleaching, mordanting, and dying was studied to know the changes in each step and to know a better combination for dyeing from them. With the results of the treated fabrics, biocompatibility tests were carried out to determine their antimicrobial effects as well as cytotoxicity. Biodegradation is an important phenomenon for any material, and to know the biodegradation pattern, soil burial methods were carried out on treated as well as untreated silk fabrics. The application of the treated silk fabrics in different fields was analyzed. Accordingly, the following objectives were set for the present investigation:

1: To study the effect of traditional washing formulations and commercial detergents on Eri, Muga, and Pat silk clothes.

2: To study the effect of curcumin on the dyeing of Eri, Muga, and Pat silk clothes.

3: To study the biocompatibility and biodegradation of Eri , $Muga$, and Pat silk clothes treated in objectives 1 and 2.

4: To develop a silk-based water filtration setup and a formulation for silk washing

1.11 Reference

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