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ADVANCES IN DEGUMMING OF MULBERRY SILK: FROM CONVENTIONAL PRACTICES TO ECO-FRIENDLY INNOVATIONS

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ABSTRACT

The traditional degumming process of silk, which involves the removal of sericin to yield lustrous fibroin fibres, typically relies on harsh chemical treatments such as soap and alkali solutions. These conventional methods, while effective but pose significant environmental and health concerns due to high water consumption, energy usage and chemical discharge. In response to growing sustainability demands, eco-friendly degumming techniques have gained increasing attention. This review explores alternative green/eco-friendly approaches including enzymatic, steam, Ultrasonic, acid degumming and the use of natural or biodegradable agents. These methods aim to minimize environmental impact while preserving silk quality and mechanical integrity. Enzymatic degumming in particular offers high specificity and efficiency under mild conditions, making it a promising for industrial-scale adoption. The paper also discusses the comparative effectiveness, cost considerations and scalability of various eco-friendly techniques. Transitioning to sustainable degumming not only supports ecological goals but also enhances the commercial appeal of silk in environmentally conscious markets.

Keywords: Degumming, Enzyme, steam, CO₂ supercritical fluid, Ultrasonic.

Introduction

Silk, often revered as the Queen of Fibers, has long been prized for its luxurious texture and lustrous appearance in the textile industry (Zhu *et al.*, 2022). However, beyond its traditional use in garments and fabrics, silk has garnered significant attention in recent years for its promising applications in the biomedical field (Freddi *et al.*, 2003).

Silk is a natural protein fibre produced by various arthropods, predominantly from the classes Arachnida (e.g., spiders and mites) and Lepidoptera (e.g., butterflies and moths). Among these, the silk derived from the domesticated silkworm *Bombyx mori* L., is the most widely utilized and commercially important (Pandey *et al.*, 2022). Structurally, silk is primarily composed of two distinct proteins i.e., fibroin and sericin. Fibroin forms the inner core of the silk fibre and is an insoluble fibrous protein responsible for the

fibre strength, elasticity and structural integrity (Chares Subash and Muthiah, 2021). Sericin, a water-soluble, globular protein that acts as a glue, binding the fibroin filaments together. These two proteins differ significantly in their appearance, solubility, amino acid composition and the presence of reactive functional groups (Nakpathom *et al.*, 2009). Due to these differences, fibroin and sericin are typically separated prior to further processing.

If sericin is not removed from silk in textile applications, it can significantly affect the quality and performance of the final fabric. The presence of sericin results in a stiff and rough texture, reducing the soft and smooth feel typically associated with high-quality silk. It also masks the natural luster of silk, giving the fabric a dull appearance (Uyen *et al.*, 2023). Additionally, sericin interferes with dye absorption, leading to poor color uptake, uneven dyeing and reduced colorfastness. Fabrics with residual sericin

have lower breathability and moisture-wicking ability, making them less comfortable to wear. Moreover, since sericin is water-soluble, it can wash out unevenly over time, leading to fabric degradation and reduced durability during laundering.

Degumming of silk refers to the process of removing sericin, a gummy protein coat that surrounds the silk fibroin fibers produced by the silkworm (Devi *et al.*, 2017). This is a vital step in the silk processing industry to produce soft, lustrous and dyeable silk fabric.

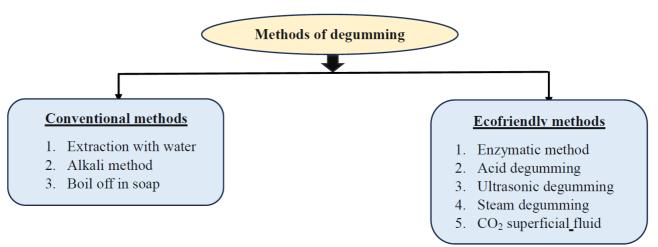


Fig.1: Methods of degumming

Conventional methods

Extraction with water

At high temperature, sericin can be removed from water. It is not a commercial process. By extracting sericin with water at 115°C for 3 hours, up to 96 percent of sericin can be extracted. Although it is still uncertain whether the removal is actually achieved through hydrolysis or dissolution (El-Sayed *et al.*, 2022).

Alkali method

Alkali degumming is less expensive than soap degumming in terms of chemicals and labour, enhanced productivity. But, it might leave silk yellowish and abrasive. As a result, alkalis should not be utilized alone.

The pH of the boiling bath should be kept between 9.5 and 10.5 when using alkalis. The pace of degumming is too sluggish below pH 9.5, while the risk of chemical damage is too severe above pH 10.5. Alkaline buffers such as sodium carbonate, sodium bicarbonate, sodium hydrogen phosphate, trisodium phosphate and potassium tetraborate, boric acid have been utilized many times. The most commonly used buffer is sodium carbonate and sodium bicarbonate (Rastogi and Kandasubramanian, 2020 and Allardyce et al., 2016)

The concentration of electrolytes in the degumming bath in addition to the buffer influences the rate of degumming. The rate rises as the alkali content rises. Degumming takes 60 minutes at a pH of 10 and a temperature of 95°C with a 0.01 molar sodium carbonate and sodium bicarbonate concentration, but only 20 minutes with a 0.05 molar alkali concentration at the same pH and temperature (Wu *et al.*, 2025).

Boil off in soap

Silk degumming has been done for almost 200 years by boiling it off in soap solutions. Marseilles soap, which is made from olive oil is the approved as a standard method. Many alternative soaps have been tried and shown to be effective. Palm-oil soap, lard-oil soap and oleic acid soap can also be used (Tsunokae, 1928).

The alkali produced by soap hydrolysis is responsible for the degumming action of soap solution. The alkali creates a chemical link with sericin, resulting in the formation of soda salt. The soap separates the swollen sericin, which then dissolves in water due to the soap's emulsification effect. The amount of soap necessary for thorough degumming is determined by the type and nature of the silk. It's an old method that appears to be still popular in the commercial world (Zhu *et al.*, 2022).

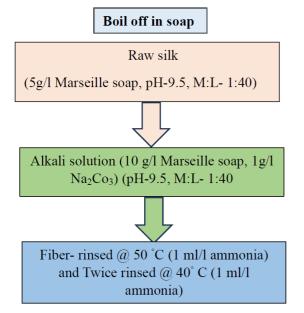


Fig. 2: Degumming by Soap Hydrolysis

Raw silk fibers were soaked for 24 hours in a solution containing 5.0 g/l of Marseille soap. The pH was adjusted to 9.5 and M:L ratio of 1:40 was used. After 24 hours, the silk fibers were immersed in an alkaline solution containing 10.0 g/l Marseille soap and 1.0 g/l sodium carbonate. The conditions maintained were 95°C for 2 hours at M:L ratio of 1:40 and pH 9.5. The fibers underwent degumming by this treatment. They were first rinsed at 50°C with 1.0 ml/l ammonia solution and twice at 40°C with 1.0 ml/l ammonia. Finally, the fibers were rinsed with cold water. (M:L-Material to liquor ratio) (Schmidt *et al.*, 2023).

- ❖ Traditional degumming methods, particularly those involving soap, hot water and alkali have long been used to remove sericin from raw silk. Although these methods are considered effective they are often associated with several notable drawbacks that limit their widespread application
- ❖ They require large amounts of water and energy, as silk must be boiled at high temperatures for extended periods often 30 to 60 minutes. This not only increases operational costs but also contributes to environmental degradation
- The wastewater generated is rich in organic matter such as dissolved sericin and soap residues, leading to high levels of biological and chemical oxygen demand (BOD and COD) in water bodies if not properly treated.
- ❖ Additionally, traditional methods can result in excessive removal of sericin, causing a substantial loss in the weight and strength of the silk fibres.

❖ Overprocessing may even damage the fibroin, affecting the lustre, softness and dye-absorption capacity of the final product. Uneven degumming is another issue, potentially leading to inconsistent texture and quality in silk fabrics. These drawbacks highlight the growing need for more sustainable and controlled processes.

In response to these challenges, eco-friendly degumming methods such as enzymatic degumming, acid degumming, ultrasonic degumming, steam degumming and CO_2 supercritical fluid have gained attention. These approaches use biodegradable enzymes and acids to selectively break down sericin without harming fibroin, offering a gentler, more sustainable alternative that reduces water consumption, energy use and environmental impact.

Ecofriendly degumming methods

Enzymatic method

Degumming of silk with enzymes is usually a two-step process. The silk is treated with a solution containing soda ash for a particular period of time in the pre-degumming stage and then further degumming is done in a solution comprising protease enzyme and non-ionic detergent in the second step. Since, they may dissolve sericin without altering the silk fiber protein, several alkaline, acidic and neutral proteases have been explored as degumming agents. Several proteases particularly alkaline proteases have been shown to remove sericin and improve silk surface qualities such as handling, shine and smoothness. Trypsin, bacterial enzymes and papain are the most common enzymes used to degum silk (Rahman *et al.*, 2020).

Papaya skin, pineapple skin and guava leaf dried by exposure under sunlight until it becomes crunchy and then crush it with mortar-shell and bring it to powder form as far as possible. Then mix these extracted powders with amount of 10 gm per 100 ml Phosphate buffer and kept in a dark place for 24 hours. Then the solution filtered and finally the enzyme extracted from Papaya skin, pineapple skin and guava. Enzyme bath was prepared by adding the 2 g Anilozyme-P, 0.5 g sodium bicarbonate, 1 g non-ionic detergent with M:L ratio 1:20.35 g of silk yarn skein washed with soft water was immersed into the prepared enzyme bath. The temperature maintained at 55°C to 60°C using incubator for a period of 100 mins. The degummed silk varn was removed followed by washing, rinsing and drying (Antony et al., 2013).

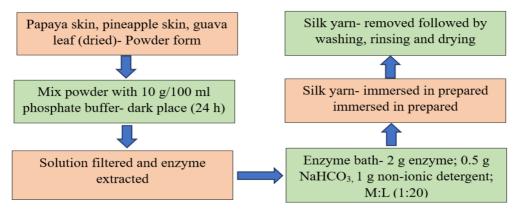


Fig. 3: Degumming by Enzymatic Methods

Acid degumming

Acid degumming effect appears to be related to the hydrolysis of proteins at certain amino acid sites. Acids target aspartic and glutamic acid residues, which are found in far greater abundance in sericin than fibroin. Although acidic degumming has received little attention, it is considered to be safer than alkaline degumming. Effective degumming occurs with acids in the pH range of 1.5 to 2 (Khan *et al.*, 2010). Silk that

has been degummed and tested with citric and tartaric acids has yielded positive results. Because mineral acids can cause significant damage to silk, only organic acids are often used. It has been discovered that weight loss is almost completely independent of acid type for various acids. Dilute organic acids, unlike alkalis, target the peptide links that connect aspartic and glutamic acid. Attacks on the above link are 100 times faster than attacks on other bonds.

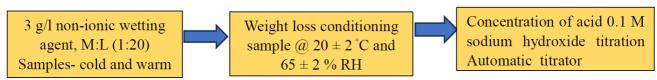


Fig. 4: Acid Degumming

Acid degumming was performed under different conditions of time, temperature and acid concentration with 3 g/l nonionic wetting agent, liquor ratio 100:l. Degummed samples were washed with cold and warm water and then dried at room temperature. Weight loss was measured after conditioning the samples at 20°C and 65% RH. The concentration of tartaric acid in the degumming bath was determined by 0.1 M sodium hydroxide titration.

Ultrasonic degumming

Great directionality, ubiquitous penetration and the ability to strengthen the diffusion, exchange and transfer of matter can help the treating fluid reach the crossing points of silk fabric and the function of ultrasonic wave can make use of acoustic cavitation effect to produce impact on sericin layer at the crossing point, thus promoting the fragmentation and separation of sericin, improving the removal efficiency of sericin at crossing points and achieving uniform silk fiber degumming (Mahmoodi *et al.*, 2012). The function of ultrasonic wave can cut down the time of process and avoid the damage of fibroin through controlling the

temperature and dose of chemical agent (Yukes et al., 2012).

The process of ultrasonic degumming was as follows

Water-bath degumming under the effect of ultrasonic wave (at 80°C for 20-40 min with liquor ratio 1:100) \rightarrow washing (at 80°C for 20 min with liquor ratio 1:100) \rightarrow drying under 105° C for 1 hour \rightarrow weighing \rightarrow working out the degumming rate (Cai *et al.*, 2012)

Steam degumming

As one kind of efficient processing methods for the biomass conversion, the used steam has higher efficiency of heat transfer due to its greater heat capacity and not decreasing the moisture content of treated objects like wood, silk compared with hot air. In the textile wet process steam treatment is often used for padding dyeing, printing and finishing process (Wang *et al.*, 2018).

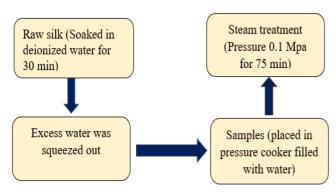


Fig. 5: Steam Degumming

Fabric samples of the same size were soaked in deionized water for 30 minutes, then the soaked samples were removed from the water bath, excess water was squeezed out and the samples were placed in a pressure cooker (XFS-280CB, a pressure meter was equipped on the cover filled with a certain volume of water. The steam treatment was carried out at a pressure of 0.1 MPa for 75 min.

CO₂ supercritical fluid

 ${\rm CO_2}$ supercritical fluid (CSCF) can be considered as the ${\rm CO_2}$ above its critical temperature (304.25 K) and critical pressure (7.38 MPa) under which ${\rm CO_2}$ shows some unique properties, such as appropriate viscosity and diffusivity like gas, appropriate density and solvating properties like liquid, making it as solvent candidate so that CSCF can be applied in many fields.

In textile field, CSCF is usually used for dyeing due to its environmentally friendly nature for the replacement of organic solvents or water, easy recovery and recycling compared with traditional dyeing process. Besides the application in dyeing synthetic or natural fibers, CSCF can also be used for pretreatment of cotton and flax fibers (Lo and Chao., 2017)

The supercritical carbon dioxide containers are heated to temperatures between 95 to 127°C. CO₂ levels are kept between 150-400 atm, for a processing time between 45 to 70 minutes Carbon dioxide emissions are removed and recycled for later use.

Table 1: Comparison of different degumming methods

Degumming methods	Advantages	Limitations
Conventional (Soap, alkali or water)	Simple processCan be applied widely	 Uses-non-recyclable chemicals Damages the fibroin Require large amount of water and energy
Enzyme	 Works under mild condition Cause minimal harm to fibroin Very specific and efficient 	 Relatively expensive Enzyme can lose activity easily
Acid	Produce smooth and clean surfaceImprove the tensile strength of silk	• Slightly lowers the ability of the silk to absorb dye
Steam	Costs lesser than other methodsDoes not require any chemicals	Not yet widely used in industry
Ultrasonic	 Increase the efficiency of degumming Reduces the amount of water and chemicals used 	 Still needs soap, alkali, acid or enzyme to work Low efficiency in converting electric energy into sound energy
CO ₂ supercritical fluid	 CO₂ can be recycled Cause little damage to fibroin 	 Needs sophisticated equipment Require acid pretreatment and ultrasonic posttreatment

Effect of different degumming methods on properties of silk

Silk fibers of *B. mori* cocoons were degummed with different concentration of citric acid (Khan *et al.*, 2010) and their physical property and fine structure were investigated to elucidate the effects of citric acid treatment. The sericin removal percentage was almost 100 % after degumming with 30 % citric acid, which resulted in a total weight loss of 25.4 % in the silk

fibers. The surface morphology of silk fiber was very smooth and fine, that showed perfect degumming like traditional soap-alkali method and tensile strength was also increased after degumming with citric acid (507 Mpa), whereas the soap-alkali method decreased the tensile strength to 250 Mpa.

Cai et al. (2012) degummed the silk in different processing duration with and without ultrasonic treatment. The results showed that the sample

degummed with ultrasonic process showed high degumming efficiency (18.43 %) than the conventional method (13.23 %). The whiteness index and fabric strength were better in ultrasonic treated sample for 90 °C at 20 min. Scanning electron microscope (SEM) images showed that there is little sericin on noncrossing places while a great quantity sericin still left at crossing points of the warps in conventional degumming process. Sericin on the crossing points is completely removed after 40 minutes of ultrasonic process and silk filament look quite smooth.

Lo and chao (2017) used an innovative CO_2 supercritical fluid method for degumming and its impact on silk fabric were examined. The SEM images indicated that the silk sample treated with CSCF had a smoother surface, better handle and texture. Additionally, the dyeing ability of the silk treated with CSCF showed highest colour strength compared to conventional method.

Degumming was performed using various enzymes extracted from natural sources such as papaya, pineapple skin, guava leaf with different enzyme concentration of 10 %, 15 % and 20 % and 35 °C, 45 °C and 55 °C temperature that influence the degumming efficiency. Among naturally degummed samples, the papaya skin enzyme shows the highest degumming efficiency (15.9 % at 15 % concentration and 45 °C temperature) while the conventional method showed 16.6 %. Tensile strength recorded higher value (661.4 N and 566.5 N) along warp and weft direction in the sample degummed with the enzyme extracted from pineapple skin compared to the conventional method (582.2 N and 467.5 N) (Rahman *et al.*, 2020)

Pan *et al.* (2024) conducted degumming of silk fibers using a combination of steam and microwave pretreatment. When microwave steam synergistic degumming was performed (10 min of microwave, 120 °C of steam temperature and 30 min of steam time), the degumming efficiency of the silk fiber was three times of the traditional method. In terms of time efficiency, the traditional Na₂CO₃ degumming took 2.75 h, while this novel method took only 0.75 h. The hygroscopicity and air permeability were also best compared to traditional treating sample.

Vyasa et al. (2016) compared the efficiencies of five different degumming treatments for eri, tasar and mulberry silk fabrics using conventional, ultrasonic and microwave techniques. Among the various treatments soda ash plus hydrogen peroxide and enzyme (papain) degumming recipies gave the best results in terms of weight loss and absorbency.

The degumming ratio, fine structure, and physical properties of silk fibers were evaluated to assess the effects of steam treatment. Unlike conventional methods using neutral soap or sodium carbonate (Na₂CO₃), steam treatment removes sericin without chemicals while maintaining desirable fiber properties. Energy and cost analyses indicate that steam degumming is more efficient and economical than traditional methods (Wang *et al.*, 2018)

Pan *et al.* (2024) developed a silk degumming method using microwave pre-treatment and steam, achieving three times the efficiency of traditional Na₂CO₃ degumming with half the energy consumption. The process took only 0.75 hours compared to 2.75 hours for the traditional method. Optimal results were obtained with 10 minutes of microwave treatment, 120°C steam for 30 minutes, yielding the highest mechanical strength: warp 542.20 N, weft 356.19 N.

The mulberry silk was degummed with commercial grade bromelain and with sodium carbonate. 96.58% of sericin content was removed from the silk yarn in small scale degumming procedure with 2 g/L bromelain and 91.84 % in large scale degumming with 5 g/L bromelain. Scanning electron micrographs of the silk yarn degummed with enzyme showed neither sign of destruction in its morphology nor surface damage. The surface of the yarn degummed with bromelain was smoother than that of the yarn degummed with sodium carbonate. (Ninpetch et al., 2015).

Conclusion

Eco-friendly degumming methods such as steam treatment, enzymatic degumming and ultrasound-assisted processes offer sustainable alternatives to traditional chemical approaches. These methods reduce environmental impact by minimizing chemical usage, lowering energy consumption and preserving the integrity of silk fibres. Among them, steam degumming stands out for its efficiency and cost-effectiveness, making it a promising solution for greener silk processing. These ecofriendly processes enable the silk industry to produce high quality silk while promoting environmental sustainability.

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