DEGUMMING OF SILK

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The process of eliminating the gum from the raw silk is known as degumming of silk. The gummy substance is the sericin which/ non filamentous. Besides sericin raw silk also contains other natural impurities namely fat, waxes, inorganic salts and coloring matter. Raw silk contains about 22-30% of sericin which is not useful to the silk for weaving process, that is why the degumming process will be done before weaving.

METHODS OF DEGUMMING

Sericin is insoluble in water, however, it can be hydrolysed wherein the long protein molecules broken down in to smaller units which can be solubilised in hot water. Hydrolysis of proteins can be carried out by treatment with alkalis and enzymes. acids are non specific and attack very vigorously on sericin and the processcan not be controlled and hence acids are not used for the process of degumming. Degumming of silk can also be done by using enzymes which attack specific amino acids of the sericin protein.

Thus there are three methods of degumming.

1 Extraction with Water

2 Treatment with alkalis

3 Digestion with enzymes

1. EXTRACTION WITH WATER

It has been shown that extraction with water at 120^0 C for 2 hours in four intervals removes sericin. It has been also observed that use of non ionic detergents (neutral soap) with water at high temperature is very effective in degumming process and also to maintain strength of silk yarn.

Eg., one kg of raw silk can be immersed in 15 It. of water containing 15 gm of detergent heated to 60^0 C in about 30 min., and then temperature was raised to 130^0 C and the treatment for a duration of 30 min. The silk is treated by this method are superior in strength and elongation when compared to untreated yarn. This method is commonly employed for the degumming process.

2. TREATMENT WITH ALKALIS

Alkalis have severe effect on the proteins. The main amino acids affected are cystine, serine, argenine *etc*.,. Since sericin is rich in serine content the initial attack of alkalis is on this amino acid. Degumming with soaps in the presence of alkalis isin practice since long time. The soap acts as degumming agent and alkalis aid the process.

Degumming of raw silk with alkalis depends up on pH, temperature and time (duration) of treatment. At higher pH the alkalis attack fibroin. The optimum pH should be 8.77. This can be obtained by using solution of sodium carbonate and sodium bicarbonate. By using this buffer and treating the silk for 2 hours at a temperature of 80-100° C, one can achieve complete removal of sericin.

3. DIGESTION WITH ENZYMES

The ability of proteolytic enzymes to hydrolyze peptide bonds formed by specific amino acids has been effectively exploited in degumming process. The enzymes are trypsin, pepsin, papain etc., of these enzymes, trypsin and papain have been recommended for degumming of silk, since both of these enzymes are specific in their action.

Use of papain: Papain is obtained by the latex of papaya tree *(Carica papaya).* This latex contains two proteolytic enzymes chymotrypsin and papain. Papain is widely studied enzyme.

Degumming of silk with papain requires a pretreatment of the material whit hot water. Subsequently the incubation of enzyme in the degumming liquor under suitable pH and temperature will be carried out with an activator such as KCN or ascorbic acid etc., .After papain degumming it is necessary to wash the silk in warm water

Procedure for Degumming:

1. Weigh the given raw silk sample and note down the weight as W_1 g.
2. Prepare the degumming bath in the ratio of 1:40 (1portion is raw silk and 40 is distilled water/ soft water)
3. Add neutral soap flakes and soda ash at the rate of 7g/l and 1g/l respectively and keep it for heating.

4. Raise the temperature to boiling level and introduce the raw silk and treat for 1 h at this temperature. Constant stirring of raw silk is necessary while working.
5. After an hour take out the material, wash it in running water thoroughly to remove the soap and soda.
6. Again keep the material in soft water and give a hot wash for 30 min to remove the traces of soap and soda.
7. After hot wash, wash the silk in running water thoroughly and dry at room temperature under shade. After complete drying, weigh the degummed silk and note down the weight asW_2 g.
8. Find out the degumming loss by using the following formulae.

Degumming loss in % = $\frac{W_1-W_2 \text{ X100}}{W_1}$ = ____ %.

Fibroin per cent age= 100 – Sericin per cent age= ___ %.

Report: The sericin and fibroin % in the given raw silk material is _____ and _____ % respectively.

Observations and Calculations:

Weight of the raw silk = _____ (W_1) gm.

Weight of the degummed silk = _____ (W_2) gm.

Liquor ratio 1:40 *i.e.*, W_1 X 40 = ---- ml of water (a)

Soap 7gm/l = $\frac{7 \text{ X a}}{1000}$ = ___ gm.

Soda 1gm/l = $\frac{1 \text{ X a}}{1000}$ = ___ gm.

Degumming loss = $\frac{W_1-W_2 \text{ X100}}{W_1}$ = ____ %.

Fibroin per cent age= 100 – Sericin per cent age = ___ %.

SILK BLEACHING

Silkworms of many varieties produce colored cocoons like yellow, green, red,pink *etc*., of various shades. The silk colour is mainly due to two pigments namely carotenoid and flavonoids. Carotenoids are distributed mainly over sericin where as flavonoids are distributed even in the fibroin in addition to sericin. So degumming also removes colouring matter that is produced by carotenoids but not by flavonoids. Therefore to get pure white silk bleaching is necessary before dyeing.

METHODS OF BLEACHING:

The bleaching process used for the bleaching of silk based on the eitherreducing agents or oxidizing agents.

The prominent reducing agents used for bleaching are

1. sulfur dioxide
2. Sodium hydro sulphide
3. Sodium or zinc sulphoxylate-formaldehyde

The oxidizing agents recommended for bleaching are

1. Potassium permanganate
2. Sodium perborate
3. Sodium peroxide
4. Hydrogen peroxide (H_2O_2)

Hydrogen peroxide is the most preferred bleaching agent. Chlorine based bleaching agents namely bleaching powder, sodium hypochlorite *etc*., are also generally used.

Hydrogen peroxide is the most preferred bleaching agent commonly used for commercial bleaching purpose. Hydrogen peroxide is sold as 35 % or 50 % aqueous solution. The reason why hydrogen peroxide acts as a bleaching agent is due to simultaneous process of activation and stabilization of its solutions. Activation favorsthe formation of a perhydroxy ion ($H0_2$-), which can be actually regarded as the true bleaching agent. Stabilization avoids the formation of molecular oxygen which can be obtained by dissociation.

1. $H_20_2 \leftrightarrow H + H0_2^-$

2. $2HO_2^- \leftrightarrow 2OH^- + O_2$

The addition of alkali to an aqueous solution of hydrogen peroxide displaces the equilibrium of reaction 1. towards the right, favouring the formation of perhydroxy ions.

$H_2O_2 + OH^- \leftrightarrow H_2O + HO_2^-$

Activated hydrogen peroxide (ion HO_2^-) is relatively unstable and has to be stabilized to limit the formation of molecular oxygen, which diminishes the efficiencyof bleaching.

Under alkaline conditions, hydrogen peroxide can be bleached at room temperature with a pH of 10.5, with sodium silicate (for stabilization of activated H_2O_2). At pH of 10.5 silk is chemically damaged a lower pH range of 8-9 is recommended. The pH adjusted either ammonia or sodium pyrophosphate.

Typical bleaching requirements are

1. 5gm / lt H_2O_2 (35%)
2. Igm / lt prestogen
3. l grn / lt ammonia (25%)

Treatment for 30 min at 70° C & wash.

Depending up on the whiteness required, the bleaching operation may be altered. Bright white silk is not always preferred. For dyeing of very light shades, it isdesirable to have double bleaching.

Optical whitening: In order to enhance the whiteness of a fabric, treatment of fabric with optical whitening agents are used. The common commercial optical whiteningagents used for silk are Ranipal WHN, Ranipal WG *etc*,.

Procedure for Bleaching:

1. Weigh the given silk sample and note down the weight as W_1 g.
2. Prepare the bleaching bath in the ratio of 1:20 (1portion is silk and 20 is distilled water/ soft water)
3. Add H_2O_2, liquid ammonia and EDTA at the rate of 20ml/l, 1g/l and 1ml/l respectively and keep it for heating.

4. Introduce the material, raise the temperature to 60°C and treat the material for 1 h at this temperature. Constant stirring is necessary while working.
5. After an hour take out the material, wash it in running water thoroughly to remove the soap and soda.
6. Again keep the material in soft water and give a hot wash for 30 min.
7. After hot wash, wash the silk in running water thoroughly and dry at room temperature under shade. After complete drying, weigh the bleached silk and note down the weight asW_2 g.
8. Find out the bleaching loss by using the following formula.

$$\text{Bleaching loss in \%} = \frac{W_1 - W_2 \times 100}{W_1}$$

Report: The bleaching loss in the given silk material is _____ %.

Observations and Calculations:

Weight of the silk = _____ (W_1) g

Weight of the bleached silk = _____ (W_2) g

Liquor ratio 1:20 *i.e.*, W_1 X 20 = ----- ml of water (a)

Volume of H_2O_2 20ml/liter = $\frac{20X\ a}{1000}$ = ___ ml.

Volume of EDTA 1gm/liter = $\frac{1\ X\ a}{1000}$ = ___ gm.

Volume of liquid ammonia 1ml/ liter = $\frac{1\ X\ a}{1000}$ = ___ ml.

Bleaching loss = $\frac{W_1 - W_2 \times 100}{W_1}$ = ___ %.

Dyeing

Dyeing is defined as an operation or a series of operations by means of which uniform colour of a permanent character is produced on a substance. This implies that it should not be possible to wash the colour in laundering nor it should fade rapidly when exposed to light.

Silk has all desirable properties of a textile fiber i.e., strength, elasticity, coolness, softness and affinity for dyes. Silk is composed of

fibroin and sericin. Presence of sericin gives dull colour to the material and also causes problem in dyeing process. Therefore, before dyeing the raw silk is generally subjected to degumming process. Usually about 22-30% of sericin, f the total weight of the raw silk s present. As sericin is removed silk becomes soft, luster and permeable for dyes.

Mechanism of Dyeing

A piece of silk cloth is composed of millions of fibroin macromolecules arranged in crystalline and amorphous forms. Different amino acids from which the fibroin is made are along the length of the molecules by peptide bonds and these macromolecules are interlinked mainly by sideways links such as hydrogen bonds. Each macromolecule has a carboxyl group (-COOH) at one end and an amino group (-NH2) at another and like an amino acid from which it is made. Under certain conditions the hydrogen ion is attached to the carboxylic group is transferred to the amino group. So that the end of the macromolecules acquire opposite charges.

When silk containing such molecules with electrically charges ends is entered into a solution of an acid, say HCl, some of the negatively charged carboxylate groups (-COO) take up hydrogen ions released from HCl into the solution and become electrically neutral carboxyl groups (-COOH) and the chloride ions also released from the HCl are absorbed and retained by the positively charged amino ends of macromolecules.

Therefore, when silk is entered into a solution of an acid in water the concentration of the acid in the solution is discharged by the absorption of some amount of the acid with the formation of an electrically positive site, whenever the amino group is present in the fibroin molecule.

The amount of acid absorbed by the fibroin increases with time until it reaches a particular value, after which it remains constant. This constant value is called the equilibrium absorption of acid by silk.

Types of Dyeing

Mainly there are four types of dyeing. They are

1. Acid Dyes
2. Basic Dyes

3. Direct Dyes
4. Reactive Dyes

1. Dyeing of silk with acid dyes

Though the silk has an affinity for acid dyes, the dyed shades tend to be les fast than wool. However, silk exerts its affinity for acid dyes a lower temperature than in the cases with wool and the dyeing is usually started at 40C and the temperature allowed to rise to about 85C. it is rarely necessary to exceed 85C and oiling is be avoided if possible since boiling exerts a deleterious effect on the tensile strength and luster of silk.

Procedure for dyeing with acid dyes:

1. Weigh the given degummed silk sample and note down the weight as W_1 g.
2. Prepare the dye bath with dye solution by taking required amount of water (*i.e.*, at the ratio of 1:40) and glauber's salt (10%).
3. Start the dyeing at room temperature and raise the temperature to 40 °C gradually. Work the material for 15 min at 40 °C.
4. After 15 min take out the material from the dye bath and add required amount of acetic acid stir the solution and again dip the material into dye bath.
5. Raise the temperature to 90-95 °C and work for about 45-60 min.
6. If the colour is not exhausted in the dyeing bath, add some additional amount (2%) of acetic acid and continue the dyeing.
7. After 45-60 min take out the material, wash it in cold water and dry under shade.

Observations and Calculations:

Weight of the degummed silk = _____ (W) g

Liquor ratio 1:40 *i.e.*, W X 40 = ------ ml of water

Glauber's salt @ 10% $= \frac{10X\ W}{100} =$ ___ gm.

Acetic acid @ 4% $= \frac{4\ X\ W}{100} =$ ____ ml.

Dye Stuff 1. Orange 1% = $\frac{1XW}{100}$ = ___ gm.

2. Green 2% = $\frac{2XW}{100}$ = ____ gm.

1. Red 3% = $\frac{3XW}{100}$ = ____ gm.

2. Dyeing of silk with basic dyes

Dyeing of protein fiber with basic dyes involves the absorption of the cations on the anionic groups created in the fiber substance. In the silk fibers, ionized carboxyl groups at the molecular chain ends provide the required anionic sites.

Some basic dyes are

1. Auromine – O,
2. Rhodomine – B and
3. Brill magenta *etc*.,

3. Dyeing of silk with direct dyes

Direct dyes (R-SO3Na) are similar to acid dyes but have planar structures. While acid dyes have non co planar structures. However, since the ionized suphonate groups of the acid dyes are mainly responsible for the formation of a strong dye-fiber ionic bond.

Some of the direct dyes are

1. Direct Scarlet – SE
2. Direct Fast Red – F
3. Direct Sky Blue –FF
4. Dura Fast Yellow - RS *etc*.,

4. Dyeing of silk with Reactive dyes

The reactive dyes produce a change in the chemical structure of all fiber. The change was irreversible and the process is extremely fast. The silk is degummed thoroughly before dyeing with reactive dyes. The reactive dyes are one either by alkaline method or acidic method.

The dyeing silk also divided in to two groups.

1. Natural dyes and 2. Synthetic dyes *eg*., direct dyes and reactive dyes.

Natural Dyes: natural dyes are obtained by the different part of the plants like stem, root, leaves etc., and also from vegetables.

Natural dyes may be

1. Mordant Dyes: Dying is done in the thread after mordanting (to bite). It was earlier believed that bleaching proves bites in to the fiber causing holes on the surface which hold the colour. The chief mordant are Alum, Crome *etc*., whose oxides affect a chemical union with the fiber and colouring matter.
2. Vat Dyes: They produce a fast colour. The advantage of these dye stuff is that they are used in powder form and a small quantity of powder produces a large quantity of colour.

Printing of silk

Printing is also described as localized dyeing. The printing process involves thefollowing factors:

1. Method of printing
2. Preparation of printing paste/print paste composition
3. Fixation of prints
4. Washing

Methods of printing of silk are mainly

i. Hand block printing

ii. Screen printing

Hand block printing

It is a slow operation. A separate block is required for each different colour in the design. Block printing continues to be popular in India particularly for specializedjobs.

Screen printing

Screen printing is a relatively simple method of printing. Printing is carried out an a flat, solid table covered with a layer of resilient felt and washable blanket. Heat for drying the printed fabric may be provided either

under the blanket or by hot air fans above the table. One screen is required for every colour in the design.

The printing process consists of forcing the viscous print paste through the open areas of the screen with a flexible synthetic rubber blade known as squeegee. Mostly, acid dyes or metal complex dyes are used in printing. Recently, some reactive dyes have also been found suitable. For getting decorative effects, pigments, kahdi and gold printing can be carried out using synthetic polymer based binders and film forming compounds. Certain auxiliary chemicals which can act as solvents, acid liberating agents and wetting agents can be added to enhance the yield of colours and quality of prints. Various types of thickening agents are used to increase the visibility of printing paste to get sharp prints. Prints are fixed generally by steaming. Fixation of pigments is done by curing in an electrically heated oven or oil heated chamber.

Generally washing of the printed material is done manually and fabric is dried in air.

Finishing of silk

The term finishing with regard to silk, generally reminds of the following properties:

Crease resistance

Anti static effect

Spot resistance (water and oil drops)

Flame retardency

Dimensional stability

Wash and wear properties.

Fabric finishing can be broadly divided into:

a) Mechanical finishing
b) Chemical finishing

The objective of mechanical finishing is to impart or improve certain desirable qualities like drape, fall, handle, feel, stiffness, weight *etc*., most of the mechanical finishes being only temporary.

The objective of chemical finishing is to impart the desired effects, such as anti crease and flame retardant qualities. The technology of imparting chemical finishes to fabrics is well developed in cotton, polyester and blends. However, the development of these finishes on silk is still in a rudimentary stage, though efforts are beign made time and again by individuals and institutions. CSTRI has also taken up studies on evolving a suitable crease resistant finish for silk.

Mechanical finishing

Some of the machines used for mechanical finishing of silk are:

Chemical padders

This is usually used along with stenter.

Stentering machine

This machine imparts dimensional stability of fabrics.

Calenders

The handle and appearance of the fabric are improved by this machine.

Horizontal screen steamers and shrinking machines

These equipments produce shrinkage and relaxation of the fabric.

Decatizing machine

Dimensional stability is increased and the fabric is smoothened by removing the creases.

Felt calendar or palmer

This machine also smoothness the silk fabric and gives it a lustrous appearance. Thisis mainly used for taffeta and serge qualities.

Breaking machine

When just calendaring is not sufficient, the breaking machine is used to impart a soft handle to the fabric. Button type and knife type of machines are available.

Tamponing machine

The tamponing machine is used for treating fabrics which will have developed roughened surface during the earlier processing operations before finshing.

Weighting

Weighting is carried out in order to compensate the loss of weight due to degumming. The original weight can be restored or even increased by treatment with chemical such as iron compounds, tin compounds and tannin. This imparts a fuller handle and better drapability.

Scrooping

Scrooping with respect to silk is a peculiar crackling sound produced when fabric isrubbed or squeezed by hand. It is imparted by dilute acetic acid or tartaric acid treatment. Lactic acid at 10g/lt for 5-10 min. is also recommended.

Crease recovery finishes

Suitable resin precondensates are used to achieve crease resistant finish. These products are such that they either react with one another or cross link with the fibroinback bone to form water insoluble products under the action of heat and catalyst.

Many different types of N-methylol derivatives of nitrogenous compounds are usedfor this purpose.

Flame retardants

It is generally established that a reaction product of polyhalogented acids having acyclic nucleus such as chlorendic acid and thiourea would impart the self extinguishing properties.

Oil repellent finishes

Water and oil repellent finishes are applied in conjuction with each other and the conventional auxiliaries needed for this effect are, waxes, metal acid salt and oxides.

- Proteins and nitrogenous compounds
- Silicones
- Fluoro chemicals

Processing of silk on India is done by following the traditional level of technology i.e., manual processing in iron or copper vats on direct source of heat under the supervision of unqualified process masters.

The inverse relationship between the size of the organization and level of technology explains the existence of traditional, inefficient technology,

where the processing is conducted either by the weavers, or by small job processor or even by weavers co-operatives societies. Conversely, efficient mechanical processing is done in large integrated processing units. The common problems that are facing integrated finishing plants today are huge unutilized capacities and lack of attempts to adopt the machines used for polyester and cotton to silk. The failure of efficient processing systems to percolate down to the lowest level is a much due to the structural inverse relationship mentioned above as due to a total lack of extension activity within the processing field.

It is possible to improve the degumming, bleaching and dyeing process through better education of supervisory staff and workers, better organization of purchase of dyes and chemical and better services for problem solving at the field level. The advanced methods in mechanical and chemical finishes could be initiated by formulating and implementing appropriate schemes for encouraging various degree of mechanization.

It may also be suggested that centralized units may be established in different parts of the country where silk weaving units are concentrated. This will certainly benefit handloom weavers.
