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## A NOVEL COOKING FORMULATION FOR IMPROVED REELABILITY & RAW SILK RECOVERY OF SILKWORM COCOONS

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### ABSTRACT

The major problem observed during the muga cocoon reeling process is the filament breaks due to improper cooking or softening of sericin joints. With highlighting the associated pessimistic attributes of conventional soda based cooking method, this work focuses on developing a new formulation that can improvise the efficiency and uniformity of muga cocoon cooking process. Subsequently, the SoPs (Standard operating procedures) of the newly developed formulation were optimized based on intensive laboratory trials in comparison to conventional soda based cooking method. It was evident from the documented results that the formulation significantly contributed to the reelability of muga cocoons by reducing breakages (upto 20-25%) during the reeling process and improving the raw silk recovery (by 6-10%). The duration of cocoon cooking drastically reduced from traditional 10-15 mins (as in case of soda based cooking) to 3-5 mins. along with uniform cooking. The cooking solution constituting the formulation could be reused effectively for 2-3 times thereby supporting the economical conservation of materials involved.

**Keywords :** Muga silk, cocoon cooking, reelability, raw silk recovery, reusable, eco-friendly, uniformity, lustre

### Introduction

Filament breaks due to improper cooking or softening of sericin joints still prevails to be a major problem in muga reeling process. More the breaks, less will be the cocoon's reelability and hence pose drastic reduction in reeling performance of the cocoons. Unlike mulberry, wild cocoons (in this study, referring to Muga) need additive chemicals to expedite the process of cooking/ softening of sericin so as to achieve effective reeling. Despite this fact, the methods used in muga cocoon cooking vary from one unit to other in absence of a well optimized process. Reelers of North-east India (particularly the state of Assam) use their practical experience and skill to get the desired amount of softness in cocoons. Usually cocoons are cooked in water containing 2.0 – 2.5 g/l sodium carbonate with material to liquor ratio of 1: 20 for duration of 15-20 min at 95°C. Duration of cooking also varies depending on the duration of cocoon storage prior to reeling. Well, cooked cocoons give raw silk recovery of 40-45% of its initial shell weight and in some cases upto 50%, but mainly depending upon the quality of cocoons and silk waste generated during reeling process. Earlier, reelability studies were conducted on muga cocoons produced during the two commercial seasons viz., Jethua & Katia by developing various pre-treatment method using vacuum permeation and tuning of cocoon cooking parameters. The outcome indicated that the pre-treating the cocoons through vacuum permeation had neutral effect on silk recovery or reel-ability properties. The nine recipes were followed for pre-treating the cocoons and their respective silk recovery % obtained are recorded in the Table 1 given below;

**Table 1:** Crop wise performance of cocoons in terms of raw silk recovery%

#	Pre-treatment type	Jethua crop (April-May)	Katia crop (Oct - Nov)
1	Soda cooking	42.22	47.33
2	Soap & Soda cooking	43.65	44.52
3	SILKPLUS	41.27	48.18
4	Pre-steaming	41.82	47.35
5	Water soaking (18hrs)	40.44	47.74
6	Water soaking (40hrs)	42.17	46.46
7	Pre-steaming & neutralization	42.37	47.75
8	Neutralization after cooking	38.92	46.65
9	Vacuum permeation	40.69	47.71

(Source: CSTRI Annual Report 2018-19)

Among the nine recipes, the reeling performance results indicated that there was no single recipe that could consistently perform better than soda based cocoon pre-treatment and therefore, none of the recipes were recommended for implementation in the field. Thus soda based cooking (including SILKPLUS) remained as the most preferred technique which is still practiced throughout. However, there are studies indicating the negative effect of sodium carbonate treatment on silk. The soda based cocoon cooking basically targets the non-covalent bonds of silk fibroin imposing derogatory effects on silk fiber by affecting its mechanical strength and fiber thickness (Yamada *et al.*, 2001; Choudhury *et al.*, 2016). Further, there is a decrease in tensile strength of the fibre post sodium carbonate treatment, indicating partial harmful damages of cross-sectional area and microstructure of core fibroin (Khan *et al.*, 2010).

In order to avoid such detrimental effects on silk quality, it is very much important to replace/improve the existing harsh pre-treatment of cocoons with novel techniques without compromising on the silk quality. Therefore, this work was carried out with an objective to discover newer alternative solutions that aims at improving the reelability & raw silk quality in muga cocoons.

**Materials and Methods**

**Materials: muga silkworm cocoons**

Muga silkworms were semi-domestically reared by feeding them on Som (*Persea bombycina*) trees. The fully matured worms post feeding were transferred into a box-type bamboo mountages stored in indoor spaces with semi-dark and well aerated conditions and were allowed to spin into cocoons. After the completion of cocoon spinning, the defective and stained cocoons were sorted out and the good cocoons were collected for the experimental study.

**Development of cooking formulation**

The formulation constitutes two components designated as Part-A and Part-B respectively. Where, Part A is a identified surfactant (powder form) and Part B is a surface activating agent (liquid form) that are mixed together in defined concentration and dissolved in a solvent (water) to obtain a solution/formulation. The surfactant acts as a suitable agent to soften the sericin joints under boiling temperatures while surface activating agent facilitates cooking process by reducing the surface tension and increasing the wet-ability of the substrate (cocoon shell). The required concentrations and cooking parameters were determined by conducting series of laboratory trials.

**Assessment of reeling performance**

The reeling tests were carried out both in single cocoon reeling (in 5 batches, each batch containing 10nos cocoons) as well as mass reeling (100 nos cocoons) tests. In each trial, the reeling performance of muga cocoons cooked in the new formulation was comparatively evaluated against soda based cooking chemical. The cocoons selected for reeling were

conditioned in hot air oven for 2 hours and their weights were recorded. Further, the cocoons were cooked, deflossed and reeled using a suitable device until no further silk could be extracted. The oven dried weights of reeling waste (palade + fibrous waste), pupal and raw silk were recorded post reeling for the comparative analyses. The reeling performances were assessed in terms of reelability, raw silk recovery%, reeling waste generated and number of breakages occurred during reeling on Epprovette (single cocoon reeling device) as well as motorized reeling cum twisting machine (mass reeling device).

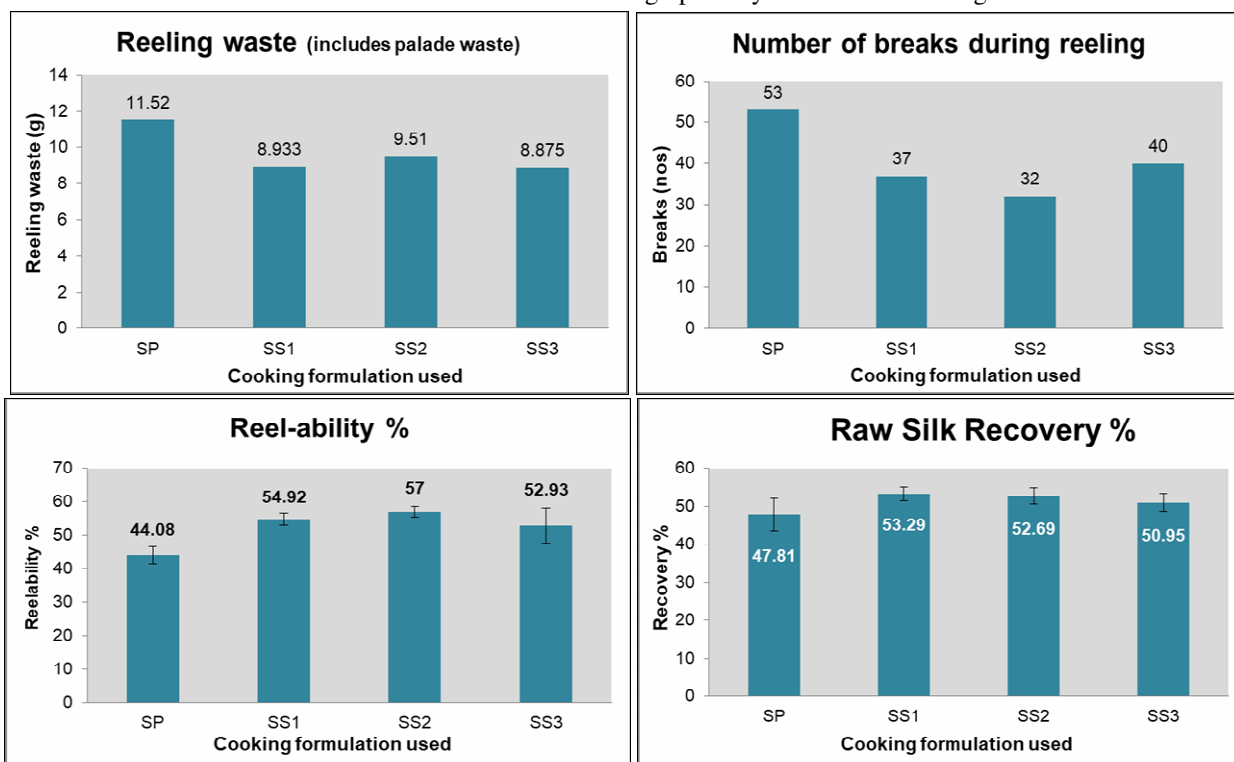
**Results and Discussion**

Trials were conducted by cooking the muga cocoons (100 nos/trial) using the mixture of Part-A and Part-B at three different concentrations designated as SS1, SS2 & SS3 respectively.

Trials	Part-A	Part-B	Preparation
SS 1	2.5 gpl	1 ml/l	The components were dissolved thoroughly in 1 litre of water to obtain a solution which was used to cook the cocoons
SS 2	2 gpl	0.8 ml/l	
SS 3	1.5 gpl	0.6 ml/l	

Cocoons under higher concentrated formulation were cooked within a very short period of 60-90 seconds were completely cooked within 60-80 seconds. Such short period gives very less room for completing the procedures involved during the cooking like soaking, stirring and lifting of the cocoons from the bath. Therefore, the concentrations and duration of cooking were optimized by keeping in mind that sufficient time is practically available to complete all the procedures involved during the cooking besides assessing their reeling performance.

The reeling parameters in terms of reeling waste generated, number of breakages occurred during reeling, reel-ability and raw silk recovery% were compared with traditional soda based cooking method (designated as SP) are graphically illustrated in the Figure 1.



**Fig. 1 :** Comparative analysis of reeling performances (reeling waste, breaks, reelability & recovery %) of cocoons cooked in soda based chemical and newly developed formulation

Based on the observations and test results, the composition of Part-A and Part-B followed in Trial 2 (SS2) was found to be optimum in cooking the cocoons effectively and giving sufficient time for handling the cocoons throughout the process. The compositions were mixed in various incremental proportions to make it up to a concentrated single formulation with a recommended dosage 40 ml of formulation in 1 liter of water for cooking of muga cocoons.

**Recommended Cooking procedure :** dissolve the composition in 1 litre of water, bring it to boil and add the cocoons. Cook the cocoons for 2-3 minutes (depending on the age of the cocoons) by completely soaking them under water. Remove the cocoons, defloss and reel.

The boil loss % of cocoon shells during cooking were calculated by following the method previously described (Nultsch *et al.*, 2018) in below equation, where  $M_{before\ cooking}$  and  $M_{after\ cooking}$  are the dry mass before and after cooking respectively;

$$Boil\ Loss\ \% = \frac{M_{before\ cooking} - M_{after\ cooking}}{M_{before\ cooking}}$$

The cocoons were cooked as per the standard procedures and the respective boil loss % calculated for both the methods are presented in the Table 2.

**Table 2 :** Comparison of Boil loss % in cooking methods

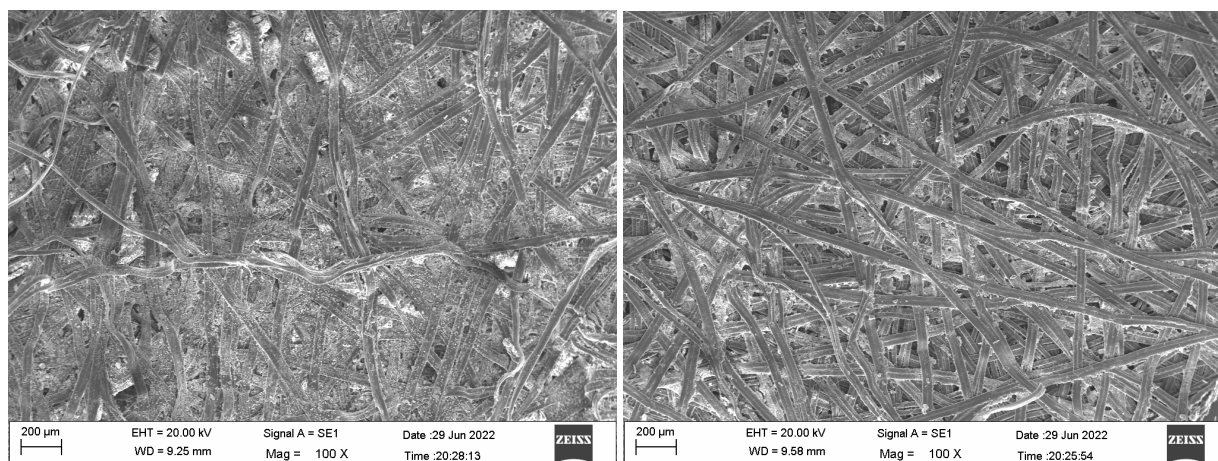
Parameter	Soda based	New Formulation
pH	11.21	9.86
Boil loss%	12.64 ± 2.23	6.48 ± 1.19

The soda based method represents higher loss % which may be attributed to the increase in severity of the treatment while the new method yielded minor loss of mass in the cooking procedures that leads positively to the inference that the formulation is being mild on the silken fibres. Previously, the effectiveness of alkaline based degumming/cooking methods and the integrity of the resulting fibroin fibers obtained were analyzed by mass loss%. Those studies conclusively reported that an excess alkaline condition poses to have a strong impact on the silk fibroin integrity and mechanical properties besides creating harsh conditions while cooking (Carissimi *et al.*, 2019). This rises the need for well controlled composition of cooking solution with optimized duration duration to reach optimum cooking of silkworm cocoons. However, the Reelers of North-east India

preferred to use their own practical experience and skill to get the desired amount of softness in cocoons without involving much of scientific approach. Thus, a milder solution that could ease the process of cooking without affecting the silk quality or any need for deviations from the traditional practices could be welcoming for easy adoption among the reelers.

An ideal cooking refers to the ability of the process to deliver uniform softening of cocoon shells right from outer cocoon shell to inner cocoon shell (palade). Unlike mulberry cocoons where, the cooking uniformity is ensured by using the technique of vacuum permeation techniques, no such sophisticated resources are available to ensure the uniformity in cooking of non-mulberry cocoons. Usually the muga cocoons are soaked in the boiling water containing soda and sauted at intervals to ensure cooking process. It is very clear that the user has no complete control over the entire cocoon lot during the cooking process without any medium of support to facilitate the through-thickness penetration of cooking solution. If the cocoons are removed for reeling soon after the outer layers are cooked, reeling becomes difficult when the middle and inner layers are reached thereby leading to increase in wastage of silk. However, the defect of under cooking is overcome to some extent by keeping the reeling water at a high temperature and longer duration, which adds to the cost of reeling besides causing other inconveniences to the reeler. The surface active agent (Part B) introduced in our formulation ensures the thorough penetration of cooking solution throughout the layers of the cocoon.

To assess the efficiency of penetration, the inner most layers of the cocoon shells obtained from both the cases of cooking were taken for microscopic observations and the resultant SEM micrographs are presented in the Figure 2. From the images it is evident that the inner surface is more efficiently cooked and appears to be uniformly reeled right from the outer surface to inner surface with reduced breakages. Such visual attributes justifies the reduction in number of breakages during the reeling process as observed in the reel-ability results. Further, the reeled silks produced from both the methods of cooking are visually compared in Figure 3. It is evident that silk reeled from the cocoons cooked under new formulation appears to more lustrous than the one produced using soda based cooking method, thus ensuring the added advantage of preventing the loss of natural luster.



**Fig. 2 :** SEM micrographs comparing the muga cocoon palades cooked using soda (left) and new formulation (right) based cooking methods



**Fig. 3 :** Visual comparison of silk yarn reeled using soda and new formulation respectively. The retention of natural luster in new method (right) can be clearly visualized

### Summary and Conclusions

The problems concerning frequent breakages and poor raw silk recovery% of the muga cocoons were established and the ways of possibilities to overcome were studied. A new cocoon cooking formulation that can facilitate efficient and uniform cooking of muga cocoons was developed and tested for its efficacy on reeling performance. Intense trials were carried out in comparison with conventional soda based cooking chemical and the comparative results were documented. The trial results indicated that the newly developed formulation significantly contributed to the reelability of muga cocoons by reducing breakages (upto 35-40%) during the reeling process and improving the raw silk recovery (by 6-10%). Based on the obtained results, an optimized procedural protocol suitable for ideal reeling process was developed. The formulation was found suitable for cooking both Muga and Eri cocoons and the cooking solution can be reused effectively for 2-3 times thereby supporting the conservation of materials involved.

Subsequently, the formulation was tested at various reeling clusters of Assam state. The test results and the feedback from the beneficiaries were promising in reducing the cooking duration and increasing the uniformity of cooking and reelability-silk recovery% of muga cocoons. The reelers have expressed their positive acceptance towards the adoptability of this formulation in achieving improved quality and quantity of raw silk. The formulation has been found suitable for Muga, Eri and Oat tasar cocoons. Awareness programs will be conducted among the reelers of major reeling clusters to adopt/popularize the newly developed cooking technique/formulation for efficient reeling and quality silk production.

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