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# CORONA TREATMENT APPLIED IN THE PROCESSING OF SILK WASTE. STUDY OF PROPERTIES SUCH AS HYDROPHILICITY AND TENSILE STRENGTH.

Daives Arakem Bergamasco[a,*], João Sinézio de Carvalho Campos[b]

[a] *College of Americana – FAM – Americana, Brasil / State University of Campinas -School of Chemical Engineering -Campinas, Brazil*
[b]*-Department of Materials Engineering and the Bioprocess- School of Chemical Engineering-State University of Campinas -Campinas,S.P., Brazil*

**Abstract**

This work makes a comparative study between the corona treatment and the traditional degumming process. For silk waste to be dyed, they must have good water absorption and mechanical strength. The results of the physical properties such as hydrophilicity and tensile strength are presented in relation to the two processes. The corona treatment promotes hydrophilicity and tensile strength compatible with the traditional degumming process, but with much less treatment time and no effluent generation.


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*Keywords:* Silk waste, corona treatment, hydrophilicity, tensile strength.

## 1. Introduction

The textile industries that promote the processing of silk waste have the function of dying silk residue substrates. But for this to happen, the substrates must have hydrophilicity, a prime factor so that the following processes, such as dyeing, can be performed. Currently the hydrophilicity of silk-based textile materials is obtained by the removal

* Corresponding author. Tel.: 55-19-983394196
*E-mail address:* daivesbergamasco@gmail.com


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of sericin, which is a natural protective layer of silk yarn, and this removal is traditionally made by washing with Marseille soap.

Corona discharge treatment promotes increased hydrophilicity in cotton fabrics without loss of the physicochemical properties of the material, and also concluded that the typical stages of primary processing (de-ing or purging) are not necessary, resulting in advantages related to Costs and processing time used in traditional processes of the textile industry [1].

This characteristic of the corona discharge surface treatment process was also tested on raw silk fabrics in the plain weave configuration where excellent hydrophilicity indexes were obtained [2].

On average, about 35% by weight of raw silk produced is waste, which is obtained in the primary (producer) and secondary (silk spinning industry) classification processes. Defective cocoons are processed and produce a thread called silk waste. Silk residues undergo primary processing such as cooking and spinning. After being spun, the yarns are ready to be woven. These yarns have a high amount of sericin, which makes it difficult to process them, so it needs to be removed by the conventional processes of degumming with Marseille soap. After degumming the silk waste yarns can be transformed into fabrics or carpets by the weaving process.

## 2. **Theoretical frameworks**

### 2.1 **Silk**

Silk is a natural textile fiber secreted by larvae of a variety of insects of the phylum Arthropoda, the main being Bombyx mori, originating from Bombyx mandarin (Theophila mandarina), commonly called silkworm. This fiber has the purpose of covering the insect and its protection during the stage of transformation of the larva into pulp, and later transformation into a butterfly [3].

The cocoon generated by Bombyx mori is composed mainly of proteins. There are three distinct proteins: fibroin, corresponding to 70-80% of the dry matter and corresponding to the main component of the textile silk yarn; Sericin (known as natural gum), in concentrations between 20-30% being the adhesive agent that holds fibroin in the conformation of the cocoon and a glycoprotein, denominated P25, in small concentrations, but with function in the maintenance of the integrity of the thread silk. Complementary components such as ash, polysaccharides and lipids can be detected [4].

Bombyx mori presents a life cycle defined in four stages: egg, larva, pupa (or chrysalis) and the final stage in the form of butterfly (called adult). The estimated life cycle for this species lasts between 55 and 60 days, depending on the breeding conditions and the type of the egg. The larvae feed on white mulberry leaves (Morus sp) for an average period of four weeks, and present great physical variations: the increase of mass from 0.45 mg to 4.5 g and its length from 3 mm to 8 cm.

#### **2.1.1 Cocoons classification**

After the cocoons are harvested in the pupa phase (before they become a butterfly) the cocoons undergo a process of cleaning and primary selection of the cocoons. The cleaning of the cocoons is done with a manual or motorized machine called a peeler or cocoon cleaner, which removes the "anafaia", that is, the poor quality and malformed silk that surrounds the cocoon.

The classification of cocoons is done in two ways:

- Quality cocoons: They are healthy, clean, uniform in color and size, without spots and with live chrysalis;
- Second quality cocoons: They are those that have small spots or defects, especially (figure 1):
  - Internal stains: caused by dead or injured caterpillars or pupae, due to harvesting done before time;
  - External stains: caused by dead caterpillars or their feces, especially when the encapsulation is uneven;
  - Peel thin: caused by weak creations;
  - Holed cocoons: caused by parasites or predators of pupae or by adult emergence;
  - Double cocoons: usually formed by two caterpillars.

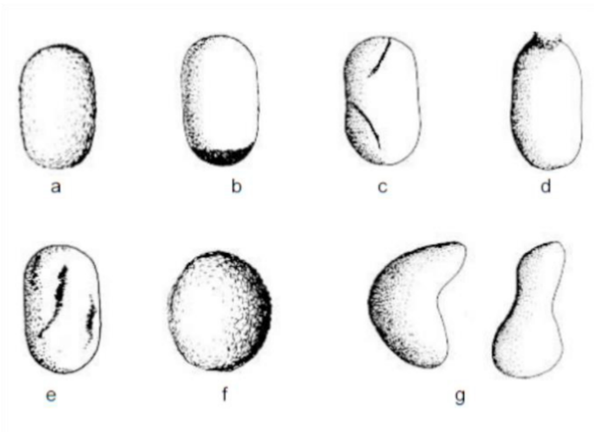


Figure 1. (a) Quality cocoons; (b) External stains; (c) Scratched; (d) Holed; (e) Dirty; (f) Double coccons; (g) irregular cocoons

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### 2.1.2 Silk Waste

On average, about 35% of the raw silk weights produced are residues, which are obtained in the primary (producer) and secondary (silk spinning industry) classification processes. Defective cocoons are processed and produce a thread called silk straw. All this material goes through a manual primary process of cooking (Figure 2 – a), so that the sericin of cocoons is softened. After the baking process, the defective cocoons go through a manual spinning process (Figure 2 – b), where the unwinding and releasing generate different characteristics for each yarn. Thereafter, the yarns are ready to be beneficiated or woven. These yarns have a high amount of sericin, which makes their processing difficult, so it needs to be removed by conventional degumming processes (figure 2 – c). After degumming the silk straw threads can be transformed into fabrics or carpets by the weaving process.

Figure 2. (a) Cooking process; (b) Manual Spinning process; (c) Conventional degumming processes

## 2.2 Corona Treatment

The corona discharge is produced in a gas (air, N2, O2,) when suitable electric potential is applied between two metal electrodes, for example a wire and a flat plate. In the region of high electric field the ionization of the gas produces active species (ions and excited molecules), which following the electric field gradient are deposited on the surface of the material (sample), which is on the plate-plane electrode of the system (Figure. 3). The effects of such active species on the tissue can be studied according to the corona parameters (voltage, current, electrode types and geometry), electrodes distance (wire/plate), atmosphere (inert or not), temperature, relative humidity and treatment time [5].

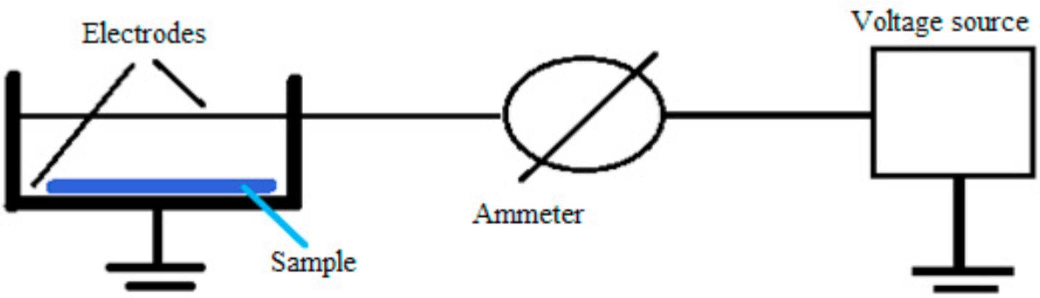


Figure 3 – Corona system

## 2.3 **Contact Angle**

One of the ways to analyze the hydrophilicity of a solid by a liquid is through the contact angle analysis. The concept of surface tension is that it defines thermodynamically the phenomenon of wettability. Upon contact with a solid flat surface, a liquid may spread completely or be in the form of a drop with a certain contact angle, see figure 4. This figure shows a drop (liquid) deposited on the surface of a material (solid) and the tangent line to determine the value of the contact angle (Φ). The Φ value varies between 0º and 180º. For Φ=0° the liquid wets the surface of the solid completely and spreads at a rate that depends on the liquid viscosity and the surface roughness. When Φ >0° the liquid does not spread completely on the surface. Thus the wettability can be assessed by the measurement of the Φ value, i.e., the lower the value of the contact angle the better is the material wettability [6]. This is easily understood because the high surface energy tends to interact more with a polar liquid, and therefore allows a greater spreading of this. For cases where the surface energy is low a drop of liquid deposited on the material surface tends to remain in the spherical shape. For non-polar liquids, the ratio is inverse, ie, higher surface tension, higher contact angle.

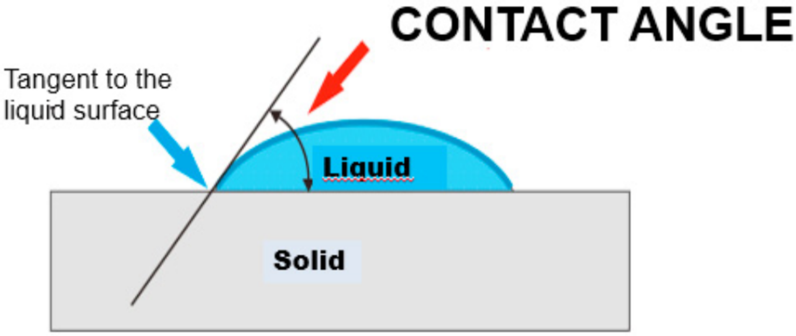


Figure 4 – Contact Angle between a drop (liquid) and a surface (solid).

## 3. Methodology

Silk waste tests were done with three sample fabric types (plain weave – 125g/m$^2$):

- raw, untreated;
- Degummed with Marseille soap;
- Treated with Corona Discharge.

The samples degummed with Marseille soap were treated in equipment like bath. The degumming process comprises treating for 1 hour at 90-95 °C, pH 9.3 to 10 for the following bath conditions: • 8-10 g / l soap from Marseille; • 1 g / l sodium carbonate;1 • g / l of sodium tripolyphosphate.

The corona treatment system (Fig.3) allows controlling the treatment time and the distance between the metallic electrodes (from 2 to 20 mm), and operates in controlled ambient temperature and humidity conditions, 27 ° C (+/- 3°) and 45% (+/- 3%), respectively. Fabric samples are trimmed with rectangular geometry (3x10 cm) and placed under the corona wire to be treated. The distance between the electrodes is previously set and the treatment time is recorded by means of a stopwatch.

Measurements of the contact angle value of a drop of water (15 µL) deposited on the treated and untreated fabric samples surfaces are carried out through a goniometer (Tantei- CAM Micro).

The fabric samples (treated and untreated) were submitted to the tensile strength tests with Texcontrol TC-10 dynamic equipment.

All measurements, contact angle and tensile strength tests, were performed at ambient conditions of temperature and relative humidity and taken in quintuplicates.

## 4. Results and Discussion

### 4.1 Contact Angle:

**Degummed with Marseille soap**

Figure 5 shows the contact angle values for 10 samples of raw silk without treatment and 10 samples degummed with Marseille soap. We can observe that the value of the contact angle for the raw samples is 100$^0$ (hydrophobic character) and for the degummed samples with Marseille soap the value is zero degree (hydrophilic character). Thus showing that degumming promotes the property of complete wettability of the material.

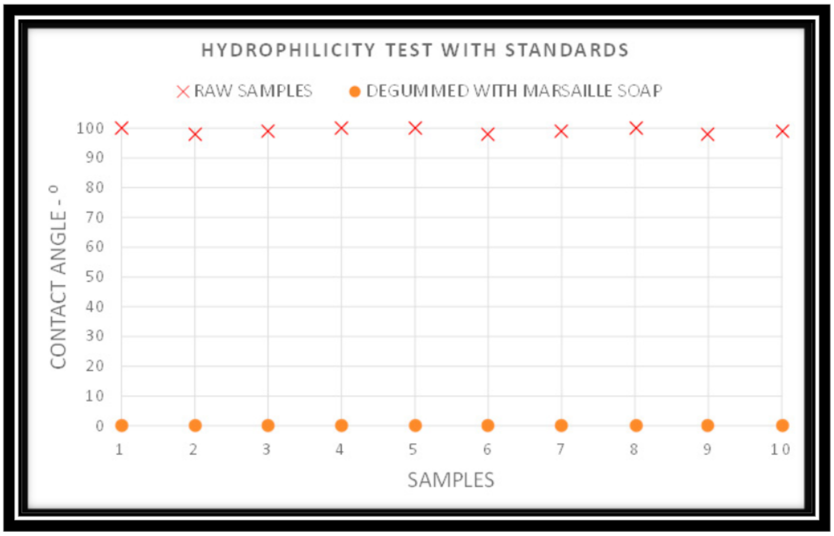


Figure 5 – Contact angle for 10 samples untreated and 10 samples degummed with Marseille soap.

**Corona Treatment**

Figure 6 shows the contact angle values as a function of time for samples treated by corona. For all distances between the corona wire and sample it is observed that contact angle tends to decrease as the corona treatment time increases. Initially the contact angle value is at $100^0$ and after 130 s of corona treatment, and independent of the distance between corona wire and sample, the value drops to zero, indicating complete wettability. Noting that for distances of 10 and 15 mm the samples had complete wettability for the treatment time of 110s.

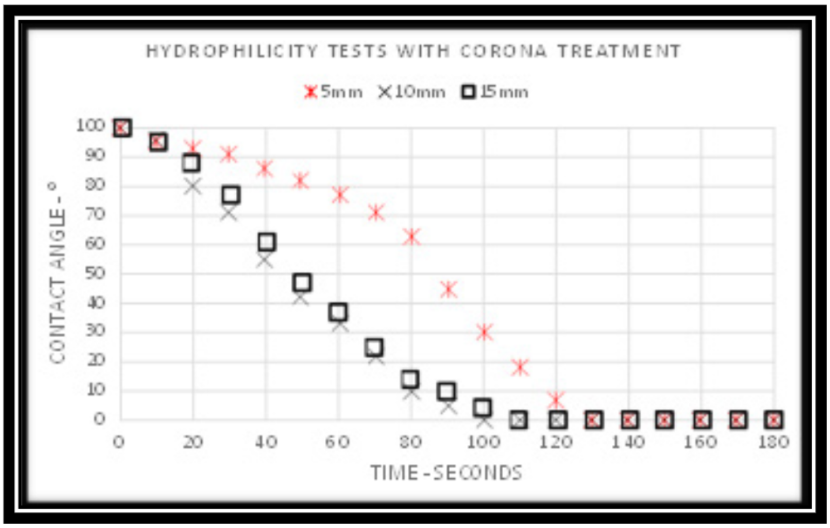


Fig. 6 – Contact angle versus corona treatment time for electrode/samples distance at 5, 10 and 15 mm.

Comparing the degumming process with the corona treatment to reach complete wettability, we have seen that the degumming time is 1 hr while 110s for corona treatment. It should also be noted that the degumming process generates a large amount of effluent. It was observed that the corona treatment is very efficient in promoting hydrophilicity and has great potential application of corona treatment in waste silk woven for subsequent dyeing.

### 4.2 Tensile strength

The results of the tensile strength tests for raw samples, samples degummed with Marseilhe soap (1h, .....) and corona treated (120 s, 10 mm) are shown in Fig.7. It is observed that the value of the tensile strength for the corona treated and degummed samples suffer a decrease in the maximum values of the tensile force in comparison with the raw samples, that is, for the raw sample the maximum tensile force is 512.5 N, for samples degummed with Marseilhe soap is 288.8 N and 411.3 N for samples treated by corona, a reduction of 43.65% and 19.74% in the tensile strength for degumming and corona treatment, respectively. This result indicates that in both cases, either by degumming or by corona treatment, the modulus of the maximum tensile strength decreases, but the waste silk fabric after corona treatment is more resistant compared to traditionally degummed tissue.

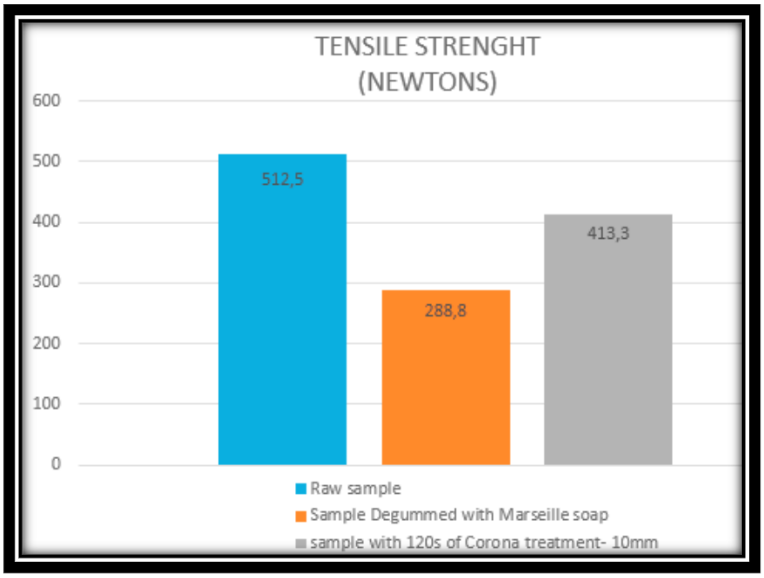


Figure 7 – Tensile strength tests for raw samples, degummed with Marseille soap and corona treated.

## 5. Conclusion

According to the tests carried out in this work, concerning the effects of degumming with Marseille soap and corona treatment on the properties analysed in waste silk fabric, it can be concluded that:

- Both degumming and corona treatment promote efficient hydrophilicity in silk waste fabric;
- The corona treatment time for complete hydrophilicity is 120 s for 10 or 15 mm distances between corona wire / fabric, which is considered very fast compared to the traditional degumming process with Marseille soap (3600 s);

- Silk fabrics treated with corona are more resistant than those degummed with Marseille soap and consequently more resistant to subsequent processes;
- The corona process does not generate effluents when compared to the degumming process.

According to these results the corona treatment has great potential of application to the processing process of the textile industry applied to residual silk fabric in comparison to the traditional process of degumming with Marseille soap.

## 6. Acknowledgements

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