SHORT TERM SCIENTIFIC MISSION (STSM) – SCIENTIFIC REPORT

The STSM applicant submits this report for approval to the STSM coordinator

Action number: FP1405 – ActInPak
STSM title: Chitosan-genipin coating: an alternative approach for active paper-based materials
STSM start and end date: 03/06/2018 to 17/06/2018
Grantee name: Gonçalo Vieira Saraiva de Oliveira

PURPOSE OF THE STSM

Chitosan-genipin films have been studied as potential materials for packaging application due to their inherent stability in acidic medium and antioxidant profile. Aiming achieving an active packaging material able to extend the product shelf-life by reducing oxidative reactions, in this STSM, the chitosan-genipin paper and cardboard coatings were studied.

DESCRIPTION OF WORK CARRIED OUT DURING THE STSMS (531)

First week started with an introduction to the bar coating technique and to the equipment for determination of mechanical properties and air permeability. Before performing any coating, mechanical properties of non-calendared paper were measured for evaluating the paper direction that offered less resistance during coating technique.

Further, a chitosan-genipin solution was prepared and applied on paper and cardboard surfaces using a blade coater machined equipped with an infrared section (Endupap) (**Figure 1**).

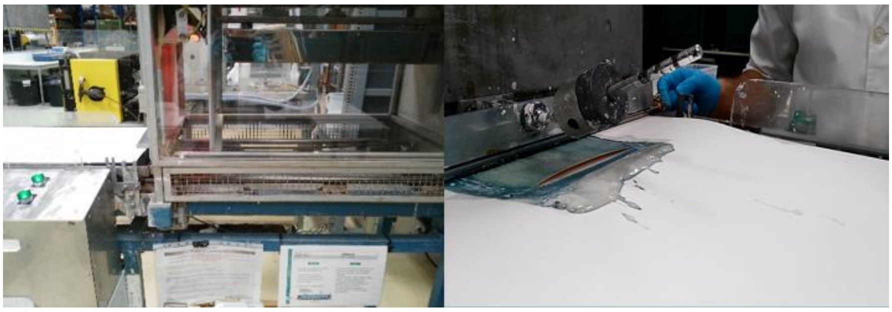

Figure 1 - Blade coater equipped with an infrared section (Endupap).

One, three and six layers of a chitosan-genipin solution were applied for paper and one, three, six, ten, twenty and thirty layers for cardboard. Two replicates were performed for each coating.

The viscosity of chitosan-genipin solution was measured immediately before and after the coatings using a Brookfield viscometer (**Figure 2**).

Figure 2 - Brookfield viscometer.

Afterwards, each coated paper and cardboard was pre-conditioned at a temperature of 23 ºC and relative humidity of 50% at least for 24 h, for subsequently characterization.

In the second week, coating process was finished and all produced coated materials were characterized. It was determined thickness, weight gain, mechanical properties (traction, burst and tear resistance) and air permeability for all non-coated and coated materials:

- Weight gain and thickness: at least 6 samples of 1.5 x 13 cm^2 from each coated material were used to determine their weight and thickness. The weight of each sample was measured using a Mettler Toledo with around 0.0001 g of precision. The thickness was measured using an Adamel Lhomargy micrometer (± 0.001 mm precision), as shown in **Figure 3**.

Figure 3 - Adamel Lhomargy micrometer.

- Traction: tensile strength, Young's modulus and elongation at break of coated and non-coated materials were determined using an Instron 5965 apparatus (**Figure 4**). Each test was performed at conditioned atmosphere (23 ºC, at 50% RH) using 1.5 x 13 cm^2 samples cut in short grain direction.

Figure 4 - Instron 5965 apparatus.

- Burst: burst index was measured using an Adamel Lhomargy EC 0.5 (**Figure 5**), according to the standard ISO 2758/2759. At least five measurements of each non-coated and coated materials were done.

Figure 5 - Adamel Lhomargy EC 0.5 used for burst index determination.

- Tear resistance: this parameter was measured using a tear tester (Noviprofibre, Elmendorf pendulum 4000mN, France), represented in **Figure 6**. Samples of 6.5 × 5 cm^2 were prepared. Only two replicates were analysed for each material due to the lack of samples.

Figure 6 - Elmendorf pendulum 4000mN used to determine tear resistance.

- Air permeability: the air permeance tests were carried out with a Mariotte vase (**Figure 7**), following the ISO 5636. A sample area of 10 cm^2 and a vacuum of 2.1 KPa were used. At least two replicates of each coated and non-coated material were performed. Only the coated surface was tested.

Figure 7 – Mariotte vase.

It was also performed an additional replicate of a 10 layers cardboard coating, with the purpose of elaborate a box model for subsequent antioxidant activity tests.

DESCRIPTION OF THE MAIN RESULTS OBTAINED

1) **Paper and cardboard coating**

The short grain direction showed lower tensile strength than long grain, thus it was the one selected for coating purposes (**Figure 8**).

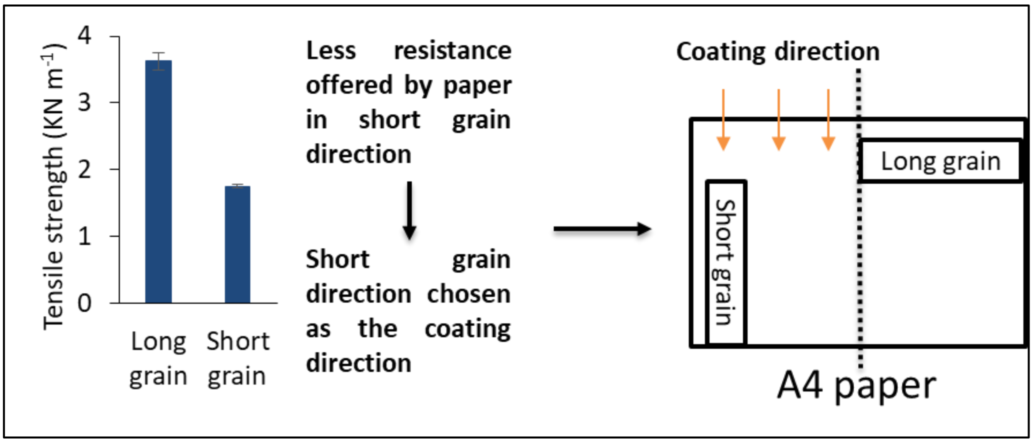

Figure 8 - Tensile strength along short and long grain paper directions and selected coating direction.

Chitosan-genipin solution was prepared by mixing 1.2% wt. of chitosan solubilized in 1.2% wt. of acetic acid solution [1] with 0.05% wt. of 10% genipin prepared in ethanol for 30 min at 50 °C. Paper and cardboard coatings were carried out using a 0.2 mm Mayer bar at 6.46 cm/s with 300 g of load. Drying step was made at IR 2500 W.

During coatings, the chitosan-genipin solution viscosity increased around 3.5 times for 1 to 20 coating layers. However, for 30 coating layers, its viscosity increased in approximately 8.5 times.

Chitosan-genipin coating promoted a greenish color on paper and cardboard surfaces whose intensity increased with the coatings number (**Figure 9**).

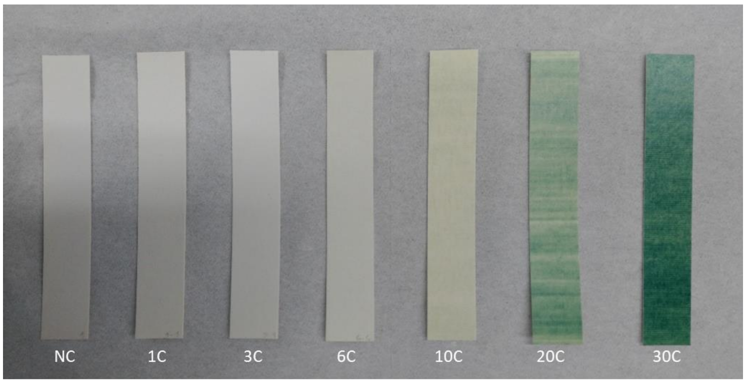

Figure 9 - Non-coated (NC) and coated (C) cardboards with different number of chitosan-genipin coating layers.

2) Characterization of the coated materials

2.1) Weight gain and thickness

Chitosan-genipin coating slightly increased the thickness of paper and cardboard for more than 1 and 10 coating layers, respectively while a significant increase was observed on cardboard with 20 and 30 layers (**Figure 10**). Only cardboards coated with 20 and 30 layers exceeded the 4% of weight gain (reference value for an acceptable coating).

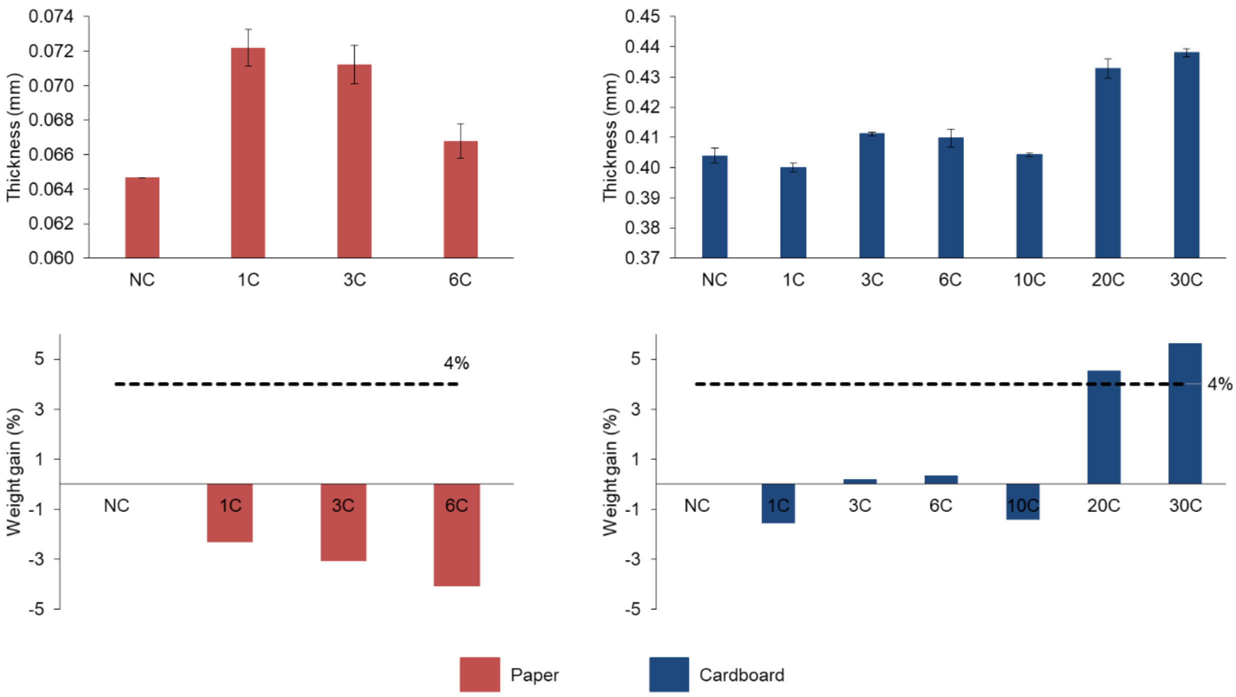

Figure 10 - Thickness and weight gain of non-coated and coated materials.

2.2) Traction tests

Chitosan-genipin solution did not affect the mechanical performance of cardboard. Contrarily, chitosan-genipin coating slightly increased the Young's Modulus and tensile strength and decreased the elongation of paper. Thus, paper became more rigid (**Figure 11**).

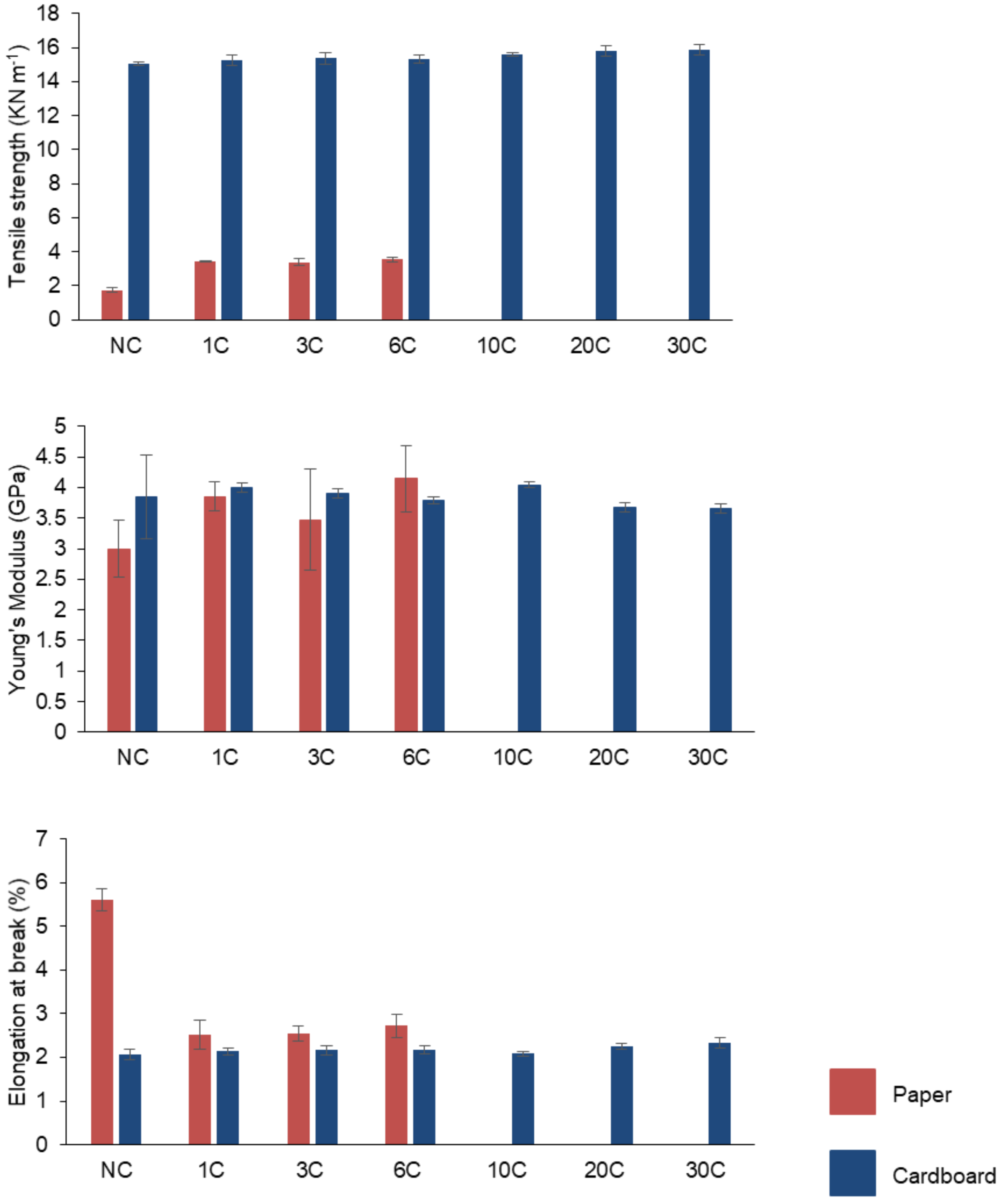

Figure 11 - Tensile strength (KN/m), Young's modulus (GPa) and elongation at break (%) of all non-coated and coated materials.

2.3) Burst

Chitosan-genipin coating slightly increased burst index for paper and cardboard, with more emphasis on paper materials (**Figure 12**). These results fit with the tensile strength results, where the highest increase observed was also for paper, which became more rigid after coating.

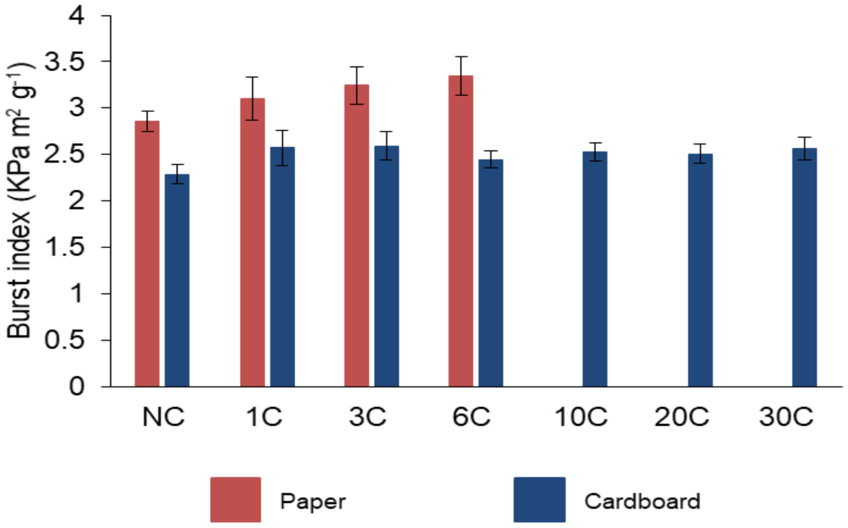

Figure 12 - Burst index results for paper and cardboard coated materials.

2.4) Tear resistance

Chitosan-genipin coating did not compromised the tear factor of paper sample and slightly increased it for cardboard (**Figure 13**).

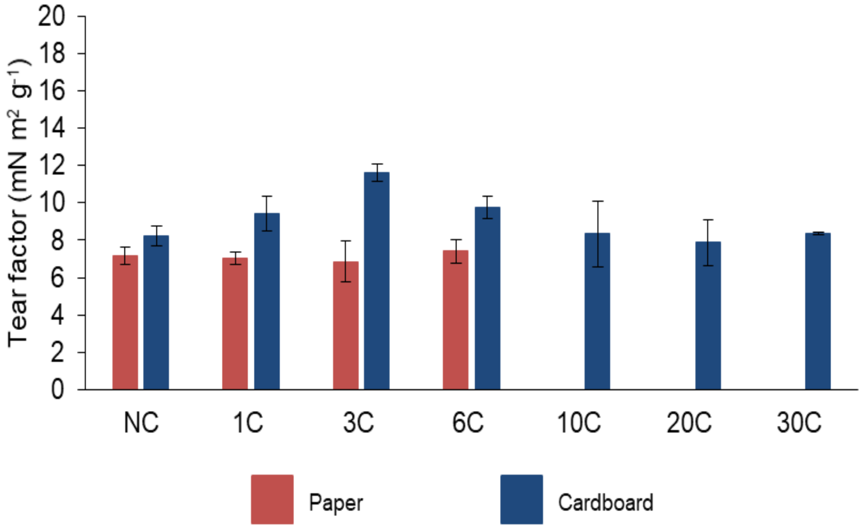

Figure 13 - Tear factors of all non-coated and coated materials.

2.5) Air permeability

Chitosan-genipin coating decreased the air permeability of both paper and cardboard. For paper, the air permeability decreased in 27% only for 6 coating layers. For cardboard, at least 40% of air permeability decrease was observed for 3 or more coating layers, attaining a maximum decrease (71%) for 20 coating layers of chitosan-genipin solution (**Figure 14**).

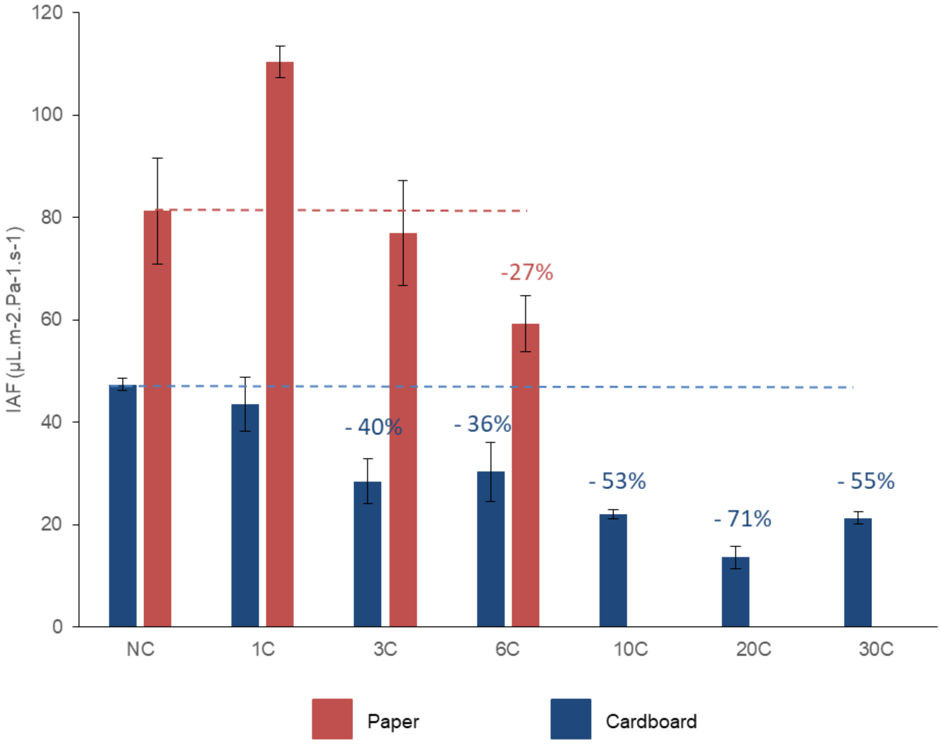

Figure 14 - Air permeability results for paper and cardboard coated materials.

[1] Kjellgren *et al.* 2006. *Carbohydr. Polym.*, 65(4): 453–460.

FUTURE COLLABORATIONS (if applicable)

N/A.