

CHITOSAN-BASED ANTIMICROBIAL LEATHER COATINGS: EVALUATION OF THE PILOT SCALE PRODUCTION



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Introduction

- ✓ Chitosan is a biopolymer that is being increasingly used in distinct areas (Fig. 1). Among its interesting biological properties, the antimicrobial activity is probably the one that generates the higher number of applications.
- ✓ Previous studies, in which leather impregnation with chitosan was performed at laboratory scale, showed that samples presented antimicrobial activity against Gram-positive and Gram-negative bacteria and hence have potential to be used for footwear components [1].
- ✓ In this work, the antimicrobial activity of chitosan-based leather coatings, produced at a pilot scale, was evaluated.

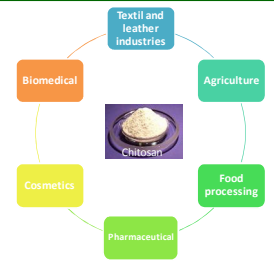


Fig 1 . Chitosan applications.

Materials and Methods

✓ **Leather coating:** Leather samples from the industrial dye fixation stage were coated with chitosan using a pilot scale drum (Fig. 2): half a hide split in two parts was impregnated during 1 hour at 50 °C. The leather was thereafter dried in industrial conditions and stored in closed plastic bags before testing. The studied process variables were:

- amount of applied chitosan:** chitosan/leather weight ratio: 0.5%, 1% and 3%;
- type of acid used to prepare chitosan solution:** formic acid and acetic acid;
- washing effect:** after impregnation, samples were divided in two pieces and only one was washed.

Antimicrobial activity:

- Test 1: **Agar Diffusion Method** based on the AATCC 147 test method [2] using *Escherichia coli* ATCC 10536;
- Test 2: **Standard Test Method under Dynamic Contact Conditions**, according to ASTM Standard E 2149-01 [3] using *E. coli* ATCC 10536. Colony counts were performed by the agar incorporation method and results expressed in colony forming units per mL (CFU/mL).



Fig 2 . Pilot drum used in the coating process

Results

Antimicrobial activity evaluation – Test 1

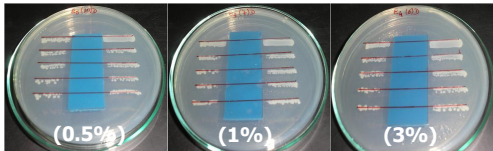


Fig. 3 Antimicrobial activity according with Test 1: leather samples coated with 0.5, 1 and 3% chitosan.

- ✓ **Test 1** was performed as a screening test for all samples.
- ✓ There were no significant differences associated with the used acid or with the existence of a washing step. Therefore, formic acid was chosen since it is already used in the industrial leather tanning process (dye fixation stage).
- ✓ Only slight differences were observed among the coated samples using different chitosan contents (Fig. 3). In view of economical costs versus an effective antimicrobial activity, 1% content was considered adequate.
- ✓ To get more accurate results Test 2 was performed.

Antimicrobial activity evaluation – Test 2

- ✓ **Test 2** was performed under dynamic contact of the sample with the inoculated solution (90 min/37 °C). Washed leather coated with 1% chitosan in formic acid and non-coated leather (control) were tested. Solution samples were collected every 15 min for colony counting. Results showed: (i) coated leather exhibited a considerable antimicrobial activity as a significant reduction of CFU/mL occurred after 15 min; (ii) no microorganisms' growth was observed at 30 min.
- ✓ **Modified Test 2** (Fig. 4) consisted in inoculum solution renewal every 15 min of testing preceded by sampling for colony counting. Results showed that, only after 5 renewals, the antimicrobial capacity is diminished (Tab. 1).

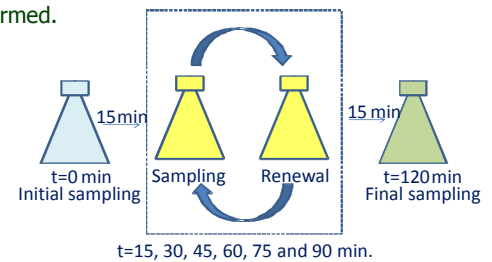


Fig. 4 Schematic representation of the sampling and renewal cycles of the inoculum solution.

Conclusions

- ✓ Comparatively with test 1, test 2 was more reliable considering the type of samples under evaluation;
- ✓ Results obtained with samples produced at a pilot scale are in good agreement with the ones obtained at laboratorial scale, evidencing antimicrobial activity according to the testing methodology applied.
- ✓ The main advantage of leather impregnation during dye fixation stage is related with the use of acidic solutions, simplifying the introduction of this new stage in the whole process.

Table 1 Results obtained in Test 2 with inoculum renewal.

Test Time (min)	Inoculum solution (UFC/ml)	Coated leather (UFC/ml)	Bacteria Reduction (%)
0	1.5 – 3.0 x 10 ⁵	1.5 -3.0 x10 ⁵	-
15	1.7 x 10 ⁵	3.1 x 10 ²	99.8
30, 45, 60, 75	1.3 x 10 ⁵ to 1.4 x 10 ⁵	0	100
90	1.2 x 10 ⁵	7.4 x 10 ²	99.5

References

- [1] M. Barros, I. Fernandes; V. Pinto; M. Ferreira; M. Barreiro; J. Amaral Proceedings of the 2nd International Conference on Biodegradable Polymers and Sustainable Composites, Nova Publishers Inc. (in press).
- [2] American Association of Textile Chemists and Colorists, *Antibacterial Activity Assessment of Textile Materials: Parallel Streak Method (1998)*.
- [3] ASTM E 2149 - 01, *Standard Test Method for Determining the Antimicrobial Activity of Immobilized Antimicrobial Agents Under Dynamic Contact Conditions*, (2001).

Acknowledges: Financial support from COMPETE, QREN and EU (project QREN-ADI-1585-ADVANCEDSHOE)

