Microcapsules Characterization on Agro Textiles

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Abstract

This paper proposes to expand a practical ecological nonwoven with antimicrobial microcapsules maintaining the constancy and biodegradability of the nonwoven for use in cultivation applications. The nonwoven was obtained using hemp fibers by Wetlaid technology. Microcapsules were prepared by co-extrusion/gelling method with alginate as shell and oregano oil as core material. The microcapsules were developed to protect and control release of oregano oil. Microcapsules were included on the nonwoven by covering method using a usual polymer as a graft substance. After incorporating microcapsules, the nonwoven was subjected to several tests in order to determinate the microcapsules fixation and their functionality. The nonwovens were characterized for their antimicrobial activity beside dissimilar kinds of bacteria and fungi. Nonwoven loaded with microcapsules was found to show good antimicrobial action in similarity with nonwoven that was not laden with microcapsules.

Keywords: *Microcapsules, Oregano Oil, Co-Extrusion and Gelling, Coating, Hemp Agrotextil and Antimicrobial Activity.*

I. INTRODUCTION

Agro textiles are a kind of manufacturing textiles used in the cultivation, horticulture, farming and production. Depending on the final function, the work, invention method and properties alter. Erosion Control Blanket (ECB) is a kind of agro textile. It is intended at intensification the soil. It is collected by a high profit of ecological natural fibers. The vegetable waste pruning can be processed by Wetlaid technology to obtain nonwoven for dissimilar applications of ECB or composites. The Wetlaid is a processing technique highly used in the paper and textile industry for nonwoven configuration. In the case of nonwoven textiles, it is possible to obtain nonwoven structures based on different base mechanism both raw and waste materials so that it is probable to combine a base natural fiber with a necessary fiber to provide unity after a thermo-bonding process. The Wetlaid process, which uses highly thinned fiber-water dispersions, is an eco-friendly process since, although it consumes high water amounts, all the water is recalculated as it only acts as the fiber delivery service component so that, almost all water is improved in the hydro former station in which nonwoven formation occurs.

The nonwoven or ECB can have an boost in its useful properties for cultivation by microcapsules integration. Microencapsulation is a technology that allows sensitive materials to be actually enveloped in a protective “wall material”, in order to defend these ingredients or “core” materials, from unfavorable reactions, volatile loss, weathering, etc.

Biopolymers, like alginate, are used as encapsulating materials in several applications, more commonly, in food and pharmaceutical applications. This polymer is not hypothetical to affect the encapsulated compound activity; it shows high toughness and it has considerable effects on the mechanical constancy of beads.

Many fundamental oils, such as garlic, oregano, cinnamon, thyme, basil, eucalyptus, rosemary, peppermint, etc., have been established to show antimicrobial activity. Therefore, these natural products are an alternative to replace synthetic chemical pesticides. The organic activity of these oils can be lost by volatilization of active components or their deprivation by act of high temperatures, oxidation and UV light. These disadvantages make the commercial application of these oils limited.

II. MATERIALS AND METHODS

A. Materials

1) Materials for Microcapsules Formation

A low thickness agonic acid sodium salt from brown algae with 3% and 5% (p/v) supplied by Sigma Aldrich (Spain) was used as shell material.

The interior material was necessary oil, oregano oil, supplied by Esencias Lozano (Spain). Calcium chloride supplied by Sigma Aldrich (Spain) was used as cross-linking agent for microcapsules formation and it was equipped at 0.5M. In order to determinate the oil by spectrophotometer was used an oil soluble dye, Verde Cornasol C (Prochimac, Switzerland), this dye was mixed with oregano oil.

*2) **Material for Hemp Nonwoven Development***

A hemp nonwoven was used as a substrate. This type of nonwoven is used as agro textile, particularly like Erosion Control Blanket. Prototypes used in the research were shaped by Materials Research Group from Textile Technology Institute. Hemp agro textile was obtained by wet-laid process with a 150 g/m2; it was composed of 80% of hemp remains complete by STW. As well as two other fibers ecological fibers those allow nonwoven to be consolidated: 10% of polylactic acid supplied by Mini fibers with 6.3 mm of length and 1.3 dpf of thickness; and 10% lyocell supplied by STW with 4.0 mm of length and 1.7 dtex of thickness.

*3) **Materials for Nonwoven Functionalisation***

An average molecular burden chitosan supplied by Sigma Aldrich was used to prepare a gel, which formed a three dimensional network that traps the microcapsules and fixed on the nonwoven. Sodium tripolyphosphate (TTP) supplied by Sigma Aldrich was used as cross linking material in order to form the polymer network on the textile substrate and stabilize the coating.

B. Methods

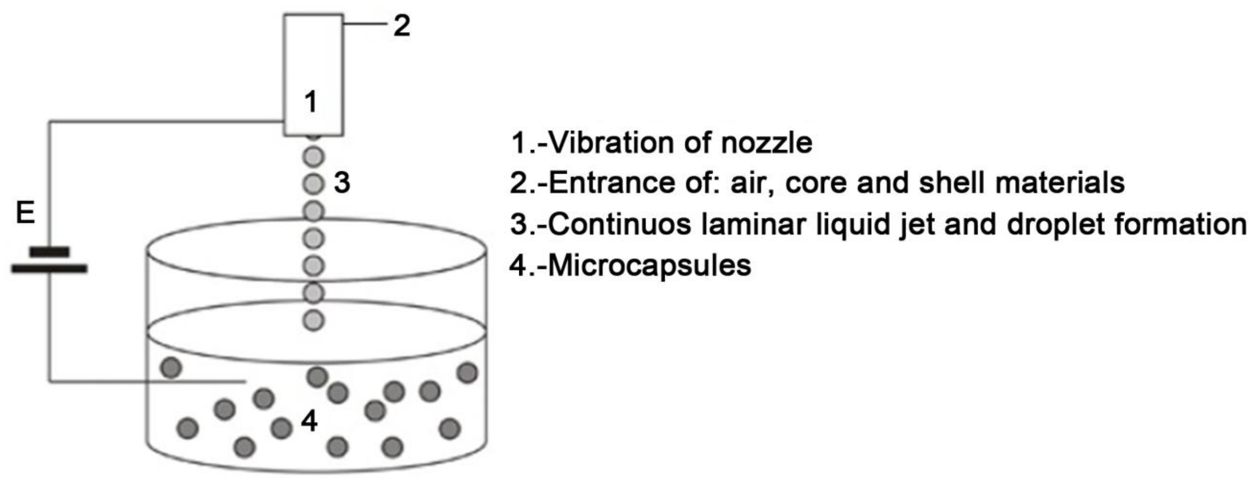

Fig 1 Microcapsule Formulation

*1) **Microencapsulation Process: Co-Extrusion/Gelling***

Microcapsules were obtained by BUCHI B-390 at room temperature. The internal nozzle diameter (core) was 0.2 mm and the external nozzle diameter (shell) was 0.4 mm. Potential and frequency values were 250 V and 350 Hz. Oil flow and alginate flow were controlled by injection pump (900 mL/h and 300 mL/h respectively), this difference is due to the solution viscosity. These experimental parameters were optimized in a previous research (Table 1).

The best parameters were selected based on:

- The final bead size;
- Final capsule size;
- The core size (amount of oil inside);
- Stability of microcapsules;
- Morphology of microcapsules.

So, microcapsules were unsoiled and washed for three times with distilled water in order to eradicate all the calcium chloride solution in the microcapsules wall (Fig.1).

	Parameters studied	Optimal parameters
Potential (V)	500, 400, 350, 300 and 250	250
Frequency (Hz)	400, 300 and 200	350
Alginate flow (mL/h)	900, 800 and 700	900
Oil Flow (mL/h)	900, 500 and 300	300

Alginate (%)	1, 2 , 2.5, 3, 3.5 and 4	3.5
Calcium chloride (M)	0.1, 0.25, 0.5 and 0.75	0.5

Table 1. The Parameters Studied And Optimal Parameters.

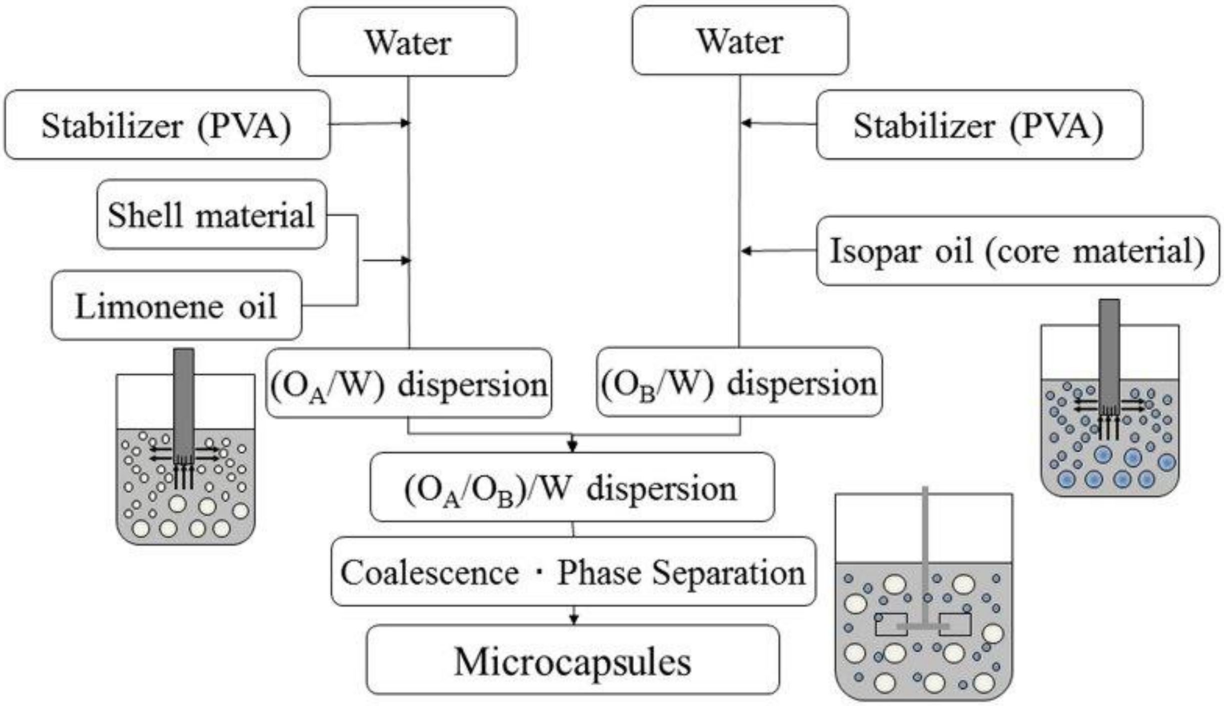

Fig 2 Microcapsules Obtaining Process

2) *Microcapsules Characterization*

Morphology:

Microcapsules morphology was considered in an optic microscope AM4115ZT Dino-Lite Edge with a Dino-Capture Software. The experiments were worked with dry and non-dry microcapsules. They were normal four times to contrast the results between different microcapsules.

Differential scanning calorimetry:

Differential scanning calorimetry Mettler-Toledo 821 was used to determinate the glass transition temperatures of the dried microcapsules. Roughly 10 mg of sample (dry microcapsules) was equipped in aluminum pans in air environment. The curves were obtained according to the following heating program from 0˚C to 220˚C at 5˚C/min.

Thermogravimetry:

Thermogravimetry was used to establish the constancy of the microcapsules at high temperatures. Around 70 µL of sample (dry microcapsules) was prepared in aluminum pans. The thermal program was located between 0˚C and 220˚C at 10˚C/min under nitrogen gas with a flow rate of 60 ml·min^{-1}.

Fourier-transform infrared spectroscopy (FTIR):

Samples were analyzed in an Infrared spectrum Varian FT-IR 670 with a 0.10 cm^{-1} resolution for each infrared spectrum. Spectra were collected in ATR mode.

III. ANTIMICROBIAL TEST RESULTS

Table 2 shows the antimicrobial test results. It compares a new nonwoven sample without chitosan gel, a sample with the chitosan gel and microcapsules. Results show that the nonwoven samples which contain the chitosan and microcapsules have a good antimicrobial behavior if they are compared with the original sample results and the samples with chitosan gel. Chitosan gel samples have a slight antimicrobial activity due to the chitosan properties, but the higher competence in antimicrobial effect is obtained thanks to the occurrence of the oregano microcapsules. This corroborates that the microcapsules endow antimicrobial properties to the nonwoven.

IV. CONCLUSIONS

Microcapsules containing oregano oil were successfully constructed by co-extrusion/ gelling using alginate as shell material. The morphology of microcapsules was spherical and the size was strong-minded by optic microscope. The formation of microcapsules established the encapsulation of oregano oil in the alginate shells by recognition of the bands and picks which corresponded with the bands in the spectra of oregano oil. Also, the antimicrobial activity of microcapsules was determined, and the results showed that the microcapsules obtainable the same behavior of free oil, therefore the encapsulation process didn't affect the antimicrobial activity of the oregano oil. Finally, thermal analysis of microcapsules shows that alginate microcapsules begin their degradation at lower temperatures than free oil. This should be taken into consideration when they are used in a procedure which conditions imply thermal treatment.

On the other hand, hemp nonwoven finished with microcapsules using a chitosan hydro gel offered good results concerning air and water permeability, and both parameters were important in agriculture to develop the soil. Also, the functional nonwoven showed antibacterial activity against different bacteria and fungi. However, more research needs to be done on stability testing and quantitative testing of antibacterial activity in different weather conditions. Lastly, this study demonstrates that the application of microencapsulated compounds on agro textiles allows functionalization and provides them with new properties.

Table.2 Antimicrobial Test Results.

Microorganism	% Reduction (cfu/mL)			
	Origin	Original Sample	chitosan sample	Sample chitosan Microcapsules
Penicillium citrinum	ATCC 1109	0.00	0.00	99.99
Rhizopus oryzae	ATCC 11145	0.00	5.00	99.99
Salmonella enterica	ATCC 14028	0.00	37.32	99.99
Escherichia coli	ATCC 25922	0.00	61.33	99.99

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