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Antibacterial and anti-inflammatory finishing of cotton by microencapsulation using three marine organisms

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ABSTRACT

This work is a small effort in the production of an eco-friendly natural based antibacterial and anti-inflammatory finished cotton fabrics using the ethanolic extracts (Ex) of the sea grass *Halophila stipulacea* (*H. stipulacea*) and marine macroalgae [*Colbomenia sinuosa* (*C. sinuosa*) and *Ulva fasciata* (*U. fasciata*)]. The extracts were phytochemically screened for their constituents. These extracts were used to finish cotton fabrics by a variety of methods. Concerning this, fabrics (F) were singly treated with ethanolic extracts (ExF) of these marine organisms by the dip technique and the extract encapsulated with sodium alginate or meypro gum. The encapsulated fabric (EnF) was further finished individually with citric acid (CA), (EnF/CA) and mono-*tert*-butyl ether of glycerol (MTBG) binder (EnF/Bin) by the pad-dry-cure technique. The fabrics so-finished were evaluated for their antibacterial and anti-inflammatory activities without washing (control) and after different washing cycles. The results obtained showed that, both EnF/CA and EnF/Bin inhibit the bacterial growth by about 90% after 10 washing cycles for both *Escherichia coli* (*E. coli*) and *Staphylococcus aureus* (*S. aureus*). The anti-inflammatory activity, the potency% reached to 88.3% for the fabric encapsulated with microcapsules of sodium alginate/*H. stipulacea* sea grass and the EnF/CA.

1. Introduction

Modern technologies have opened vast areas of research for the extraction of biomedical compounds from oceans and seas. Concerning this, intensive research for several decades has proved that marine organisms are magnificent source of a cocktail of bioactive secondary metabolites which have been reported to possess in vitro and in vivo anti-infective activity and may be directly utilized as drugs or serve as lead structures for drug development [1–5]. Structures exhibited by these natural products range from acyclic entities with a linear chain of complex polycyclic molecules and included bioactive terpenes, phenolic compounds, polysaccharides, fatty acids and alkaloids [6–8]. Interest in natural product for numerous uses is attributed to their completely different bioactivities, low toxicity and environmental property. Supported these attributes, alternative non-biological applications of natural product from marine organisms are reported to be used in textiles [9–13]. Finishing of textiles via plant extracts, in general, and marine organisms, in particular, have become a matter of significant importance because of the increased environmental awareness in order to avoid some hazardous synthetic finishes. The plant-based textile finishes with superior characteristics, especially for medical clothes are greatly appreciated and the fast development in the field of medicinal materials and their end uses has created numerous open doors for the use of anti-inflammatory as well as antimicrobial finishes [14–17]. One major problem associated with the plant extract-based textile finishing is that they are not durable.

There are diverse approaches endeavour to augment the durability and one such way is that the employ of microencapsulation. The latter is a growing technology and finds more prominent usefulness in textiles in recent years. Ipseity of microencapsulation is the littlest of coated particles and it gives a way of packaging, separating and storing materials on a minute scale for later release under controlled conditions [18,19].

This work reports for the first time on microencapsulation of antimicrobial and anti-inflammatory-rich fractions from marine macroalgae *Ulva fasciata*, *Colbomenia sinuosa* and the sea grass *Halophila stipulacea* in sodium alginate or meypro gum as coating agents. These selective species of marine organisms were identified

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and screened for their phytochemicals. For comparison purposes, the fabrics were finished by a variety of treating methods. An extensive study was conducted to assess the antibacterial and anti-inflammatory activities of the finished fabrics. The fabrics were subjected to washing durability test to find the durability of the finish and the findings are discussed in this paper.

2. Materials and methods

2.1. Materials

U. fasciata, *C. sinuosa* and *H. stipulacea* were collected from along the Abo-Qire coast, Alexandria, Cairo, Egypt, in March, May and July, respectively in two consecutive years 2013 and 2014. These marine organism samples were perfectly cleaned with fresh tap water and the epiphytes were completely removed. The cleaned samples were shade-dried at room temperature and well ground. Plain fabrics, (100% cotton woven fabric), were kindly provided by El-Mahalla El-Kubra Company for Spinning and Weaving. Meypro gum Np-16 was from Meyhall Chemical A.G., Switzerland. Sodium alginate was supplied by Sigma–Aldrich (USA). Egyptol, a nonionic detergent, was provided by the Egyptian Company for Starch, Yeast and Detergents, Alexandria, Egypt. Mono-*tert*-butyl ether of glycerol (MTBG) binder was supplied by Tetrahedron Scientific Inc., China. Other chemicals, including citric acid, carrageenan, indomethacin cream and ethanol were laboratory grade chemicals.

2.2. Preparation of the extracts

5 g of the dry powder of each marine organism were thoroughly extracted with 100 mL of 70% ethanol at 60 °C and then the ethanolic extracts were separately filtered and kept for farther treatments.

2.3. Chemical analysis of the extracts

2.3.1. Phytochemical screening

Preliminary phytochemical screening (colour reaction) was operated on the ethanolic extract according to standard methods [20–22].

2.3.2. GC–MS analysis

GC/MS is an instrumental technique by which complex mixtures of organic semi-volatile and volatile compounds may be separated, identified and quantified. Quantitative estimation of the ethanolic extract of the three studied marine organisms was achieved by GC/MS, Model (Varian 240-MS), using a capillary column VF-5, MS (30 m × 0.25 mm, ID, 0.25 μm film thickness) and a flow rate of helium as the carrier gas at 13 psi; oven temperature 50–280 °C; ion source temperature 220 °C; ionization voltage 70 eV; accelerated voltage 2000 V; volume injected 1 μL; chart speed 0.5 cm/min. The results are recorded in Table 3. The identification of the separated compounds was achieved by comparing their mass spectral data and retention times with those of the NIST (Nat. Inst. St. Technol., USA), library (Wiley Int. USA), and/or published data [23].

2.4. Finishing of marine extracts on cotton fabric

To prepare the marine extract treated fabric (ExF), the samples were given a primary wash with distilled water, air-dried and then used for finishing by the dipping method. The fabric was then immersed in the marine extract for 30 min, air-dried and then used for bioactivities's evaluation.

2.5. Microencapsulation of marine extracts

Marine extracts prepared were encapsulated adopting an emulsification technique. The apparatus employed in this technique was very simple, consisting of thermostatically controlled water bath, stirrer, a flat-bottomed glass vessel. In this technique, 10 g of each wall material (meypro gum or sodium alginate) was acquiesced to swell for 30 min of mixing with 100 mL of water. To this mixture, 50 mL of hot water was added, stirred for 15 min marinating the temperature between 40 °C and 50 °C. Core material 1% (1.5 mL of the ethanol extract of each organism) was added and stirred at 300–500 rpm for further 15 min.

2.6. Finishing of microcapsules on cotton fabric

Cotton fabric samples were padded with the aqueous solutions (10%) of the prepared microcapsules to attain a wet pick up about 100%, dried and then cured at 100–120 °C for 2 min. In order to fix the microcapsules on the fabric, the encapsulated fabric (EnF) was post-treated by two ways: (a) it was padded in 2% citric acid (CA), then dried at 80 °C and cured at 120 °C for 2 min. This post-CA-finished encapsulated fabric is referred to as EnF/CA, and (b) it was padded in 1% binder (print fix binder MTBG liquid) then dried at 80 °C and cured at 120 °C for 2 min. This post-binder-finished encapsulated fabric is referred to as EnF/Bin.

2.7. Analysis of microcapsule surface morphology

Microcapsule surface morphology was examined with a light microscope (Confocal laser scanning microscope-LSM 710 Germany) at a magnification of 400. The scanning electron microscope was used to confirm the binding of microcapsules and alignment on the fabric sample.

2.8. Antibacterial activity test

The antibacterial properties of the treated (i.e. ExF, EnF, EnF/CA & EnF/Bin) as well as untreated cotton fabrics were evaluated quantitatively (bacterial count) according to the modified AATCC test method 100–1999 [24] by using two types of bacteria, namely *Escherichia coli* (*E. coli*) ATCC 2666 gram negative (−ve) and *Staphylococcus aureus* (*S. aureus*) ATCC 6538 gram positive (+ve).

All fabric samples were kept at controlled temperature 35 °C. After incubation the fabrics were then transferred into 100 mL of nutrient broth (1:500) and shaken vigorously for 1 min. A 10-fold dilution with 0.9% (w/v) normal saline solution was prepared, spread at varying dilution onto mannitol salt agar plates for *E. coli* and maconky salt agar for *S. aureus*. Incubation to all plates was done at 37 °C for 24 h. All experiments were performed in triplicate. The antimicrobial activity is expressed in% reduction of the organisms after contacting with the test specimen compared to the number of the organism cells surviving after contacting with the control.

Bacteria reduction was calculated as follows:

$$\text{Reduction rate } \% = \frac{B - A}{B} \times 100$$

where B is the number of bacterial colonies of the untreated cotton and A is the number of colonies after 24 h contact with the treated cotton.

2.9. Wash durability of finished fabric

All fabric samples, including ExF, EnF, EnF/CA & EnF/Bin were analysed for their wash durability by subjecting the samples to washing at 40 °C for 20 min per each washing cycle by industrial