FUNCTIONALISATION OF POLYESTER FABRIC WITH BIO-BASED ANTIBACTERIAL ORTHO-VANILLIN USING MICROWAVES

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ABSTRACT (10 pt)

In the present work, ortho-vanillin was used to produce an antibacterial multifunctional polyester woven fabric using a one-shot diffusion process with microwaves.
Since the bio-based ortho-vanillin has a solubility parameter close to that of PET, it can diffuse inside polyester fibers. Two methods were investigated and compared to functionalizee polyester fabric with O.vanillin using diffusion processes: an exhaustion procedure at High Temperature and Pressure-HTHP with a liquor ratio LR 1/20 at 130°C and dip/microwave technique was used with LR 1/1 and varying radiation powers (125W, 250W, 450W) for 3 to 7 minutes.

Key Words: O-vanillin, polyester, microwave, exhaustion at HTHP, coloration

1. INTRODUCTION

Textiles today are mainly made antibacterial using products such as silver nanoparticles [1] and QACs - Quaternary ammonium compounds [2], which are being targeted due to environmental concerns. Many bio-based compounds, such as natural dyes [3,4] have already shown to impart several functionalities to textiles in addition to coloration, for example antibacterial or UV protection. Bio-based active agents from essential oils, example o-vanillin from vanilla also exhibit antibacterial activity against a broad range of microorganisms [5].
O-vanillin is a small molecule (152 g/mol), with a solubility parameter (23.8 $MPa^{1/2}$) [6] close to that of polyester–PET fabrics (21.4 $MPa^{1/2}$) -table 1. Our previous work showed that ortho-vanillin can act as carrier to improve dye diffusion inside PET fiber during polyester fabric dyeing [7].This means that, it can be potentially used to functionalize polyester fabrics using diffusion process generally employed for dyeing polyester fabrics [8].
HTHP exhaustion technique used to enhance diffusion of dye molecules polyester fabrics requires generally high pressure and temperature (130°C), a liquor ratio LR of 1/20, and use of surfactants. The huge amount of water and energy consumed for diffusion and drying processes, in HTHP dyeing, is not environmentally friendly and it is important to investigate the use of microwaves to diffuse O-vanillin inside polyester fiber. In microwave processing, energy is supplied by an electromagnetic field directly to the material. This results in rapid heating throughout the material thickness with reduced thermal gradients. Volumetric heating can also reduce processing times and save energy [9,10]. While most researchers have used microwaves for exhaustion process using higher LR ratio, in this study, we have performed a dip/microwave process to study the diffusion of O-vanillin in polyester fiber.
Both exhaustion using HTHP dyeing method and the dip/microwaves, were investigated. Spectral curves of the functionalized fabrics were analyzed using spectrophotometer, before and after removal of physi-sorbed vanillin using ethanol.

2. MATERIALS AND METHODS

2.1 PET Fabric

A 100% polyester (PET- poly (ethylene terephtalate) twill woven fabric of density 180 g/m² was used. The polyester fabric was cleaned to remove impurities using Soxhlet method with petrol ether, then with ethanol. Then the samples were rinsed in three different water-baths with distilled water, before being dried and ready for use.

2.2 Bio-based ortho-vanillin

O-vanillin is the most important component of vanilla aroma. It is a bright yellow powder obtained from Sigma Aldrich. The chemical formula, the CAS number, the vapor pressure at 25°C, and other data were obtained from an open chemistry database (Pubchem web site), see table 1.

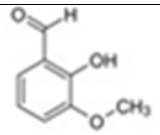
Chemical product	**Natural origin**	**Chemical structure**	**Color**	**Hildebrand solubility parameter**	**Molecular weight**	**Vapor Pressure**
CAS number: 148-53-8	Vanilla		Bright yellow	23.8 $MPa^{1/2}$	152 g/mol	6. 10^{-3} mm Hg at 25°C

Table 1: Characteristics of ortho-vanillin used in this study.

Solubility parameters $MPa^{1/2}$	δ_d	δ_p	δ_h	δ_t **(Hildebrand)**
Ortho-Vanillin	19.4	9.8	11.2	23.8
PET	18.2	7.3	7.9	21.4

Table 2: Hansen solubility parameters of ortho-vanillin and polyester-PET

2.3 Diffusion method by exhaustion procedure at HTHP

The procedures were performed in accordance with the general dyeing method using the diffusion method in a HTHP (High Pressure and High Temperature/Beaker Dyeing Machines, at 130°C) using 1 % ethanol-water solution and a liquor ratio of 1:20. The samples, weighing 5g, were treated in 200ml beakers (Labomat machine) with 1, 3, 5 and 10% o.w.f O-vanillin. The time-temperature diagram of the dyeing process is shown in figure 1a.

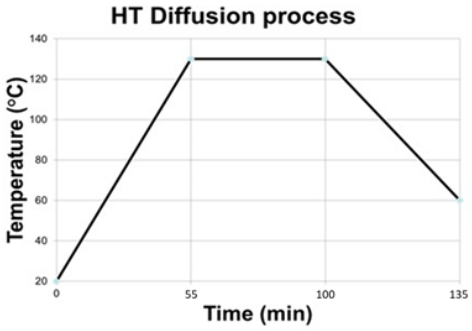


Figure 1a: Time-temperature diagram of the HTHP process

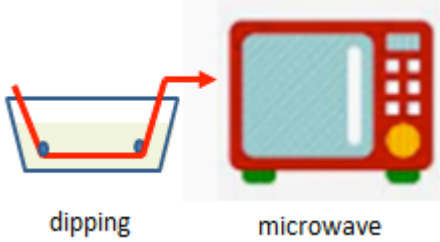


Figure 1b: Dip/microwave process

The temperature of the exhaustion bath was gradually raised (about 2°C/min) to 130°C, and was kept at this temperature for about 45 min. The bath was then cooled to 60°C; then the

fabric was squeezed, rinsed thoroughly with hot water and air dried. No surfactant was used in addition in the diffusion method bath.

2.3 Diffusion method using microwaves

Dip/microwave process (figure 1b): Samples of cleaned polyester fabric sample (12 cm x 12 cm) was dipped in a solution of vanillin (0.7 g O-vanillin, 3 ml of ethanol and 5 ml of distilled water). Fixation by diffusion using microwave was carried out in a microwave oven operating at 2450 MHz. The following output radiation powers (125W, 250W, 450W) were used during 3, 5 and 7 minutes.
The diffusion of O-vanillin inside polyester fibers induced notable pale yellow coloration of the fabric which was characterized spectrophotometric analysis.

All the functionalized samples were subjected to washing in ethanol during three minutes at 30°C to remove all particles physi-sorbed at the textile surface.

2.4 Spectrophotometric analysis of fabric samples

Reflectance of the functionalized samples "R" was measured with a Konica-Minolta CM3610A spectrophotometer for wavelength-λ varying from 360 nm to 700nm. Relative color strengths "K/S" were automatically calculated from the reflectance values by the software using the Kubelka-Munk equation (1) (Kerkeni, May 2011). K/S value is directly related to the color yield of the fabrics:

$$\frac{K}{S}(\lambda) = \frac{(1 - R(\lambda))^2}{2R(\lambda)} \quad (1)$$

where, K refers to coefficient of absorption, S is the coefficient of scatter, and R is fractional reflectance.

3. EXPERIMENTAL RESULTS

3.1. Visual and spectral analysis of the functionalized polyester fabrics

Using diffusion method by exhaustion procedure at HTHP

Diffusion of vanillin inside polyester fibers induces yellow coloration of the fabric. Figure 2a represents K/S spectral curves of the samples functionalized with 1, 3, 5 and 10% o.w.f O-vanillin;
Figure 2b shows the K/S (at λ= 400 nm-yellow) values of each functionalized PET using the different concentrations of O-vanillin. Higher K/S values of the functionalized PET fabric with increased O-vanillin (owf %) confirmed its increase uptake when its concentration in the water bath increases.

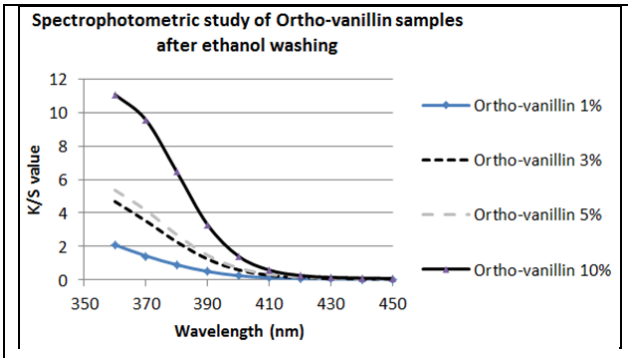


Figure 2a: K/S spectral fabrics of O-vanillin functionalized fabrics using HTHP

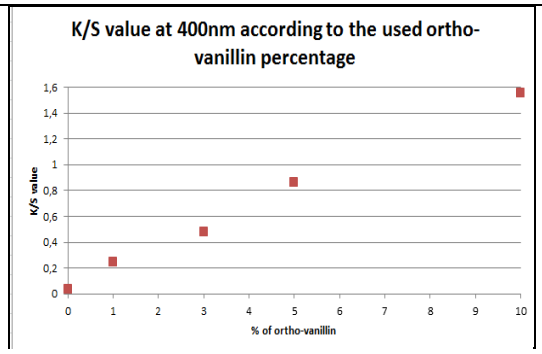


Figure 2b: K/S values at 400 nm for O-vanillin functionalized fabrics using HTHP

Using diffusion method by microwave

With dipping in O-vanillin ethanol/water solution, wet pick-up rate of the rectangular 12x12 cm fabric (2.80 g) was 100%, meaning that approximately, 8.75 % owf of vanillin was absorbed by the polyester fabric.

Figures 3 a, b, and c show the spectral curves of all fabrics subjected to various powers and duration of micro-wave treatment, after removal of all physi-sorbed vanillin using ethanol. The red curve is that of a polyester fabric with 10% o.wf vanillin using HTHP diffusion method.

When an electrical power of 125W is used, microwave duration time increases vanillin uptake by polyester fiber. K/S value at 360 nm, with a 7 minute treatment is the highest and reaches K/S similar to that using HTHP diffusion method with 5% o.wf vanillin (see figure 2a).

With the microwave process, at 250 W and above 3 minutes, vanillin uptake exceeds that of that of 10% o.w.f vanillin using HTHP diffusion method. Above an electrical power of 250W, K/S values are much higher at both wavelengths (390 nm and 400 nm), indicating an enhancement of vanillin uptake by the polyester fiber (figure 3d)

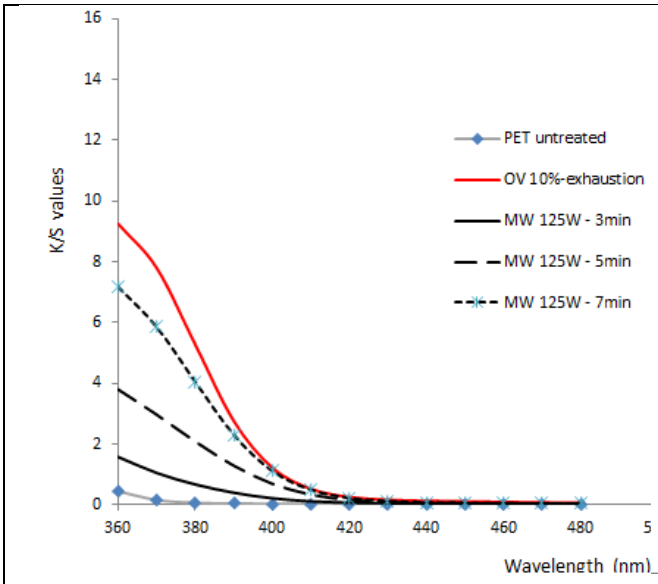


Figure 3a : Spectral curves of polyester fabric with vanillin solution subjected to microwaves at 125 W

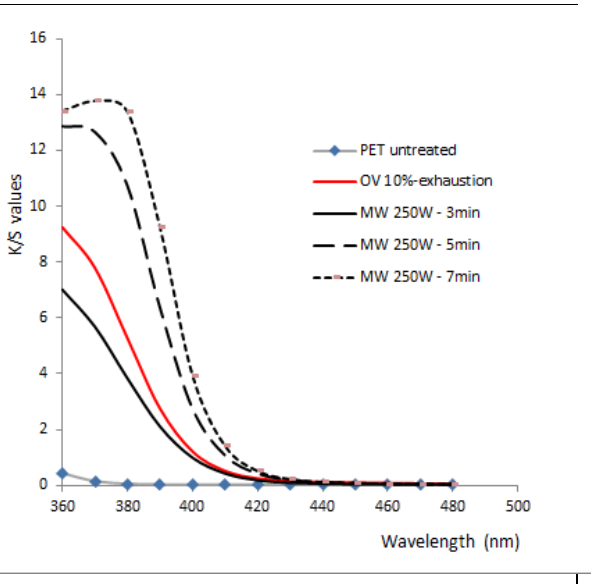


Figure 3b : Spectral curves of polyester fabric with vanillin solution subjected to microwaves at 250 W, after removal of physi-sorbed vanillin

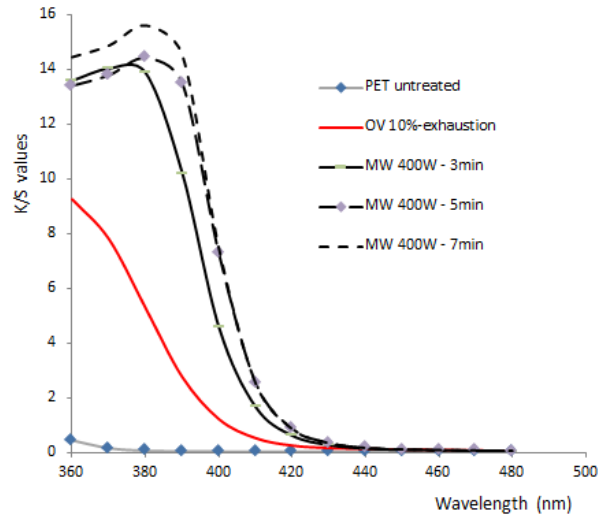


Figure 3c Spectral curves of polyester fabric with vanillin solution subjected to microwaves at 400 W

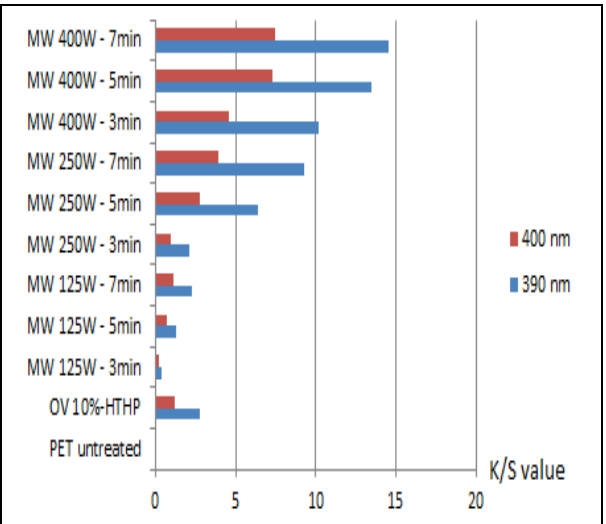


Figure 3d : K/S at 390 and 400 nm of fabrics with vanillin solution subjected to microwaves at 125W, 250W and 400 W

3.2. Antibacterial quantitative results

The sample treated with 10% of o-vanillin was tested to evaluate its efficiency against two different bacteria using the quantitative test (ISO20743:2003). The functionalized sample had very good antibacterial properties against both *Klebsiella pneumoniae* (A=4.7) and *Staphylococcus Aureus* (A=2.5).

4. DISCUSSION AND CONCLUSION

Since the bio-based ortho-Vanillin has a solubility parameter close to that of PET, it can diffuse inside polyester fibers. Two methods were investigated and compared to functionalise polyester fabric with O.vanillin using diffusion processes, using an HTHP exhaustion procedure and dip/microwave technique. Higher vanillin uptake was achieved using the microwave process. Functionalization of polyester fabrics with ortho-vanillin yielded a yellow fabric with high antibacterial activity against *Staphyloccocus aureus* and *Klebsiella pneumoniae* when 10% owf vanillin was used. Lower liquor ratio (LR1/1) and lower thermal/electrical energy used for the microwave compared to exhaustion method(LR1/20) represent a definite asset from an environmental point of view.

5. ACKNOWLEDGEMENTS

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