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Enzymatic coating of cotton with poly (ethylene glutarate)

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ABSTRACT

The present work reports the coating of cotton fabrics with poly (ethylene glutarate) for the first time. Cotton fabric were soaked in diethyl glutarate and ethylene glycol diacetate without addition of extra solvent. The reactions were catalyzed by lipase from *Thermomyces lanuginosus* at 40 °C in water bath during 7 h. The polyester coating was extracted and analyzed by MALDI-TOF and NMR. Monomers, dimers and trimers of poly (ethylene glutarate) were found. The modified cotton fabrics showed better hydrophobicity and an improvement in wrinkle recovery when compared with the untreated materials.

1. Introduction

Formation of very specific and complex chemical compounds is required to follow a sustainable industrial chemistry, as well the environmental friendly concept of green chemistry [1–3]. From this perspective, the application of enzymes as catalysts is a promising alternative to conventional industrial chemistry [4–6]. Lipases are one of the most used enzymes with a broad specificity and are widely reported for biocatalysis [7].

Lipases are used in food manufacture [8–10], in energy (biodiesel) production [7], [11,12] or in medicine industry [13,14]. Lipases are also described as bio-catalysts in a range of solvents [15,16], and emulsions [17,18]. Nevertheless a solvent-free enzymatic system can offer better processing conditions without further complex purification process [19–21].

Hydrophobic cellulosic fabrics are in great demand due to the specific properties that they endow a materials such as water repellency, self-cleaning, friction reduction and antifouling [22]. In the context of textile industry, surface modifications of cotton with long hydrophobic molecules would also improve the textile hand perception of consumers and the crease recovery, considered to be an added-value.

Despite its hydrophilicity, cotton cellulose has superb merits as a substrate for the production of hydrophobic materials: its abundance, biodegradability and unique physical, chemical and mechanical properties when compared to non-renewable materials that are classically used [22]. The hydrophobic modification of cotton cellulose has involved surface modification using hydrophobic compounds, such as fluorocarbons [23], silicones [24] and hydrocarbons [22]. However, these technologies are constricted by their sustainability and environmental concerns and cost. Finding ecologically greener alternatives to current practice is urgently needed, either chemical or enzymatic [25].

In a previous work, we have synthetized poly (ethylene glutarate) using immobilzed lipase B from *Candida Antarctica* [26]. In this paper, the soluble lipase from *Thermomyces lanuginosus* was used for the coating of cotton fabrics with poly (ethylene glutarate). The *in situ* polyesterification reaction and modification of cotton was performed in the absence of solvents. The extraction liquids of the treated cotton fabrics were analyzed by MALDI-TOF mass spectrometry and NMR spectrometry. The wettability and crease recovery performances of modified cotton fabrics were characterized to study the effect of *in situ* fabric modification.

With this work we intend to obtain hydrophobic cellulosic fabrics at good costs while using a sustainable and environmental alternative to the actual cotton modification processes.

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2. Materials and methods

2.1. Materials

The properties of the bleached cotton fabric used in this work were: 388/360 ends/picks per 10 cm and 46 g m^{-2}. Lipase from *Thermomyces lanuginosus* with an activity of over 100,000 U/g was purchased in Sigma-Aldrich, Chemie GmbH., Spain. Diethyl glutarate (DG) (purity ≥99%), ethylene glycol diacetate (EGD) (purity 99%), chloroform (AR) were obtained from Sigma-Aldrich, Co., Sintra, Portugal. All the chemicals and enzymes were used directly as received from the supplier without any further modification.

2.2. Enzymatic modification of cotton fabrics

Bleached cotton fabric was washed for 1 h with distilled water at 50 °C, dried at room temperature, and then conditioned in the standard atmosphere for at least 48 h prior to the experiments. Each cotton sample (500.0 ± 0.1 mg) was firstly soaked with 900 μl of reagent mixture, which was constituted by diethyl glutarate and ethylene glycol diacetate with the molar ratio of 1:1. About 1221U of lipase from *Thermomyces lanuginosus* with a specific activity of 407 μmol/ml/min against *p*-nitrophenylbutyrate (*p*-NPB) substrate were subsequently dripped into the cotton samples to a final enzyme loading of 25% (v/v). The pre-prepared cotton samples were placed into round bottom flasks. Reactions were carried out in water bath (WB) (OLS 200, Grant Instruments (Cambridge) Ltd., England) at 40 °C for 7 h. A reaction time of 7 h is reasonable for textile process by fed-batch. Untreated cotton fabric and cotton fabric incubated with the reagents but without enzyme were used as control. All the samples were prepared in triplicate.

After the enzymatic treatment, all the cotton samples were washed twice with chloroform to remove the remaining reagents at the surface of cotton fabric. Afterwards samples were placed inside an extractor hood at room temperature for 8 h until solvent evaporated completely.

2.3. Soxhlet's extraction of the in situ synthesized poly (ethylene glutarate)

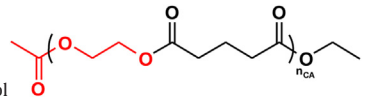
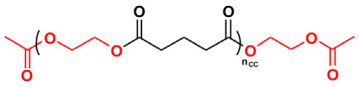
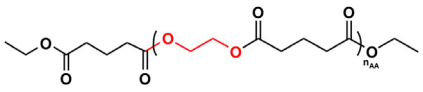
To determine the degree of polymerization of the *in situ* synthesized poly (ethylene glutarate), 150 mg of treated cotton fabrics were extracted in 80 ml of chloroform at 85 °C by Soxhlet's extraction method in Soxhlet's extractor (SXT-06, Shanghai Hongji Instrument Co., LTD, China) [27]. The extraction liquors were analyzed by MALDI-TOF and NMR.

2.4. MALDI-TOF and NMR characterization of the extraction liquid from modified cotton fabrics

MALDI-TOF mass spectra of the extraction liquid were acquired using an ultrafleXtreme MALDI-TOF/TOF mass spectrometry (Bruker Daltonics GmbH, Germany) equipped with a 337-nm nitrogen laser. The matrix, 2, 5-dihydroxy-benzoic acid (DHB) at 20 mg/ml, was prepared in a solution of 10% EtOH and 1 mM NaCl solution, and then mixed with samples (v/v, 1:1). A volume of 2 μl of each sample/matrix mixture were deposited on a ground steel target plate (Bruker part n° 209519) and then allowed to dry at room temperature in air. The dried sample spots were analyzed by the positive-ion method of RP700-3500 in the reflective mode.

NMR spectra of synthesized poly (ethylene glutarate) from the extraction liquid dissolved in 500 μl deuterated chloroform ($CDCl_3$) were recorded using a Bruker avance III400 NMR spectrometer (Bruker Corporation, Germany) at 400 MHz and 25 °C.

Table 1
Possible chemical structures of poly (ethylene glutarate) present on cotton fabric surface resulting from the lipase-catalyzed solvent free polyesterification reaction in water bath (WB) for 7 h at 40 °C with an enzyme loading of 25% (v/v)[a].

Possible end groups	Possible chemical structures
Carboxylic acid and alcohol	
Carboxylic acids	
Alcohols	

[a] n_{CA} represents carboxylic acid and alcohol as end groups of poly (ethylene glutarate). n_{CC} represents carboxylic acids as end groups of poly (ethylene glutarate). n_{AA} represents alcohols as end groups of poly (ethylene glutarate).

2.5. Contact angle measurement of cotton fabrics

Static contact angle measurement of the modified cotton was performed at room temperature in a JC2000D4 device (Shanghai Zhongchen Digital Technology Apparatus Co. Ltd, China). All the samples were kept in the standard atmosphere at room temperature (20 °C) and 65% of relative humidity for at least 48 h prior to the testing. The contact angles were measured by depositing ultrapure water drops (10 μl) on the sample surface. All the measurements were made in triplicate.

Minor amounts of bromophenol blue dye were dissolved in distilled water. A drop of 5 μl of the water with blue dye was dripped to the surface of the treated cotton samples for better visualization of the effect of modification on cotton wettability.

2.6. Wrinkle recovery measurement of cotton fabrics

To measure the effect of *in situ* formation of poly (ethylene glutarate) at the surface of cotton on the wearing comfort, wrinkle recovery angle tests were carried out using the dynamic tester of the fabric crease recovery performance (JN-1, Institute of Spinning and Weaving Technology, Jiangnan University, China). Samples were preconditioned in standard atmosphere at the temperature of 21 ± 1 °C and relative humidity of 65 ± 2% for at least 24 h [28]. All the samples were cut into the size of 40 cm × 15 cm according to the crease recovery test method [29] with their long dimension parallel to the warp direction of the cotton fabric. A pressure of 0.08 MPa was applied in all measurements. Samples were done in triplicate.

3. Results and discussion

3.1. MALDI-TOF mass spectra and NMR analysis of the extraction liquid from modified cotton fabrics

Lipase-catalyzed solvent-free synthesis of poly (ethylene glutarate) at the surface of cotton fabrics was performed using diethyl glutarate and ethylene glycol diacetate as starting reagents with a molar ratio of 1:1 and 25% (v/v) of lipase. The possible chemical structures of the formed polyester depicted in Table 1 were obtained according to the chemical structures of the starting reagents diethyl glutarate and ethylene glycol diacetate (Table 2).

The MALDI-TOF mass spectra of the extraction liquids from treated cotton fabrics are shown in Fig. 1. The polyesterification reaction yielded products with a broad molecular weight distribution from 200 Da to 1300 Da.

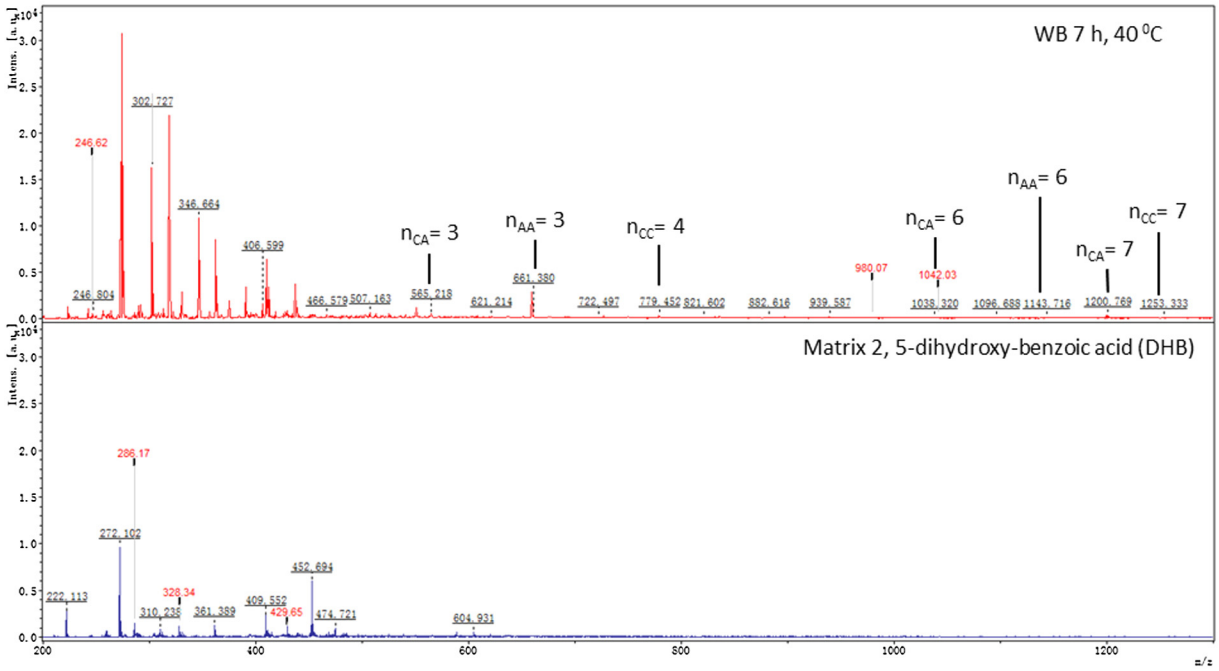
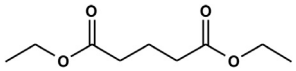
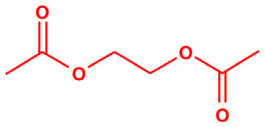


Fig. 1. MALDI-TOF mass spectra of Soxhlet's extraction liquids from cotton samples. The cotton fabrics were treated by a lipase-catalyzed solvent free polyesterification reaction in water bath (WB) for 7 h at 40 °C with an enzyme loading of 25% (v/v)[1].
[1] n_{CA} represents carboxylic acid and alcohol as end groups of poly (ethylene glutarate). n_{CC} represents carboxylic acids as end groups of poly (ethylene glutarate). n_{AA} represents alcohols as end groups of poly (ethylene glutarate).

Table 2
Chemical structures of diethyl glutarate and ethylene glycol diacetate as starting reagents for the treatment of cotton fabrics by a lipase-catalyzed solvent free polyesterification reaction.

Starting reagents	Chemical structures
Diethyl glutarate	
Ethylene glycol diacetate	

The oligomeric structures indicated in the spectra resulted from different degrees of polymerization. In Table 3 are present the degree of polymerization of the synthesized poly (ethylene glutarate) and the respective molecular weights depending on the polymers' end groups.

According to the mass spectrum (Fig. 1), the maximum degree of polymerization achieved for the synthesized poly (ethylene glutarate) at the surface of cotton fabrics was 7 (n_{CC} = 7). The presence of oligomeric structures on the extraction liquids from treated cotton fabrics indicated that the synthesized oligomers of poly (ethylene glutarate) were coated at the surface of cotton fabrics. However, any linkage of the oligomers of poly (ethylene glutarate) to the glycoside units of cotton cellulose were not detected.

NMR results showed an average degree of polymerization of polyester around 2 (Fig. 2), indicating that a high amount of unreacted components were present at the surface of cotton treated after 7 h in water bath at 40 °C.

Both results, obtained by the MALDI-TOF mass spectra (Fig. 1) and the NMR spectrum (Fig. 2), indicated that the majority of the oligomers present at the surface of cotton fabric had relatively lower degree of polymerization, ranging from monomers to trimers. The low degree of polymerization could be related with the presence of water in the initial reaction mixture.

Despite the presence of 25% of water (v/v) in the intial reaction mixture, which usually inhibits transesterification, the reaction took place resulting in the formation of several oligomeric products. A soluble lipase was used to coat the fibre because the enzyme in a solid support enzyme would not promote *in situ* polymerization [26] and [30].

3.2. Hydrophobicity analysis of cotton fabrics

Cotton fabrics possess excellent hydrophilicity when compared with the traditional non-renewable materials [22]. The hydropho-

Table 3
Molecular weight of oligomers present on the Soxhlet's extraction liquids from treated cotton samples [a]. The extraction liquids were analyzed by MALDI-TOF.

Degree of polymerization (n_{CA}, n_{CC}, n_{AA})	Carboxylic acid and alcoho l/Da (n_{CA})	Carboxylic acids/Da (n_{CC})	Alcohols/Da (n_{AA})
1	246	304	346
2	404	462	504
3	562	620	662
4	720	778	820
5	878	936	978
6	1036	1094	1136
7	1194	1252	1294

[a] n_{CA} represents carboxylic acid and alcohol as end groups of poly (ethylene glutarate). n_{CC} represents carboxylic acids as end groups of poly (ethylene glutarate). n_{AA} represents alcohols as end groups of poly (ethylene glutarate).

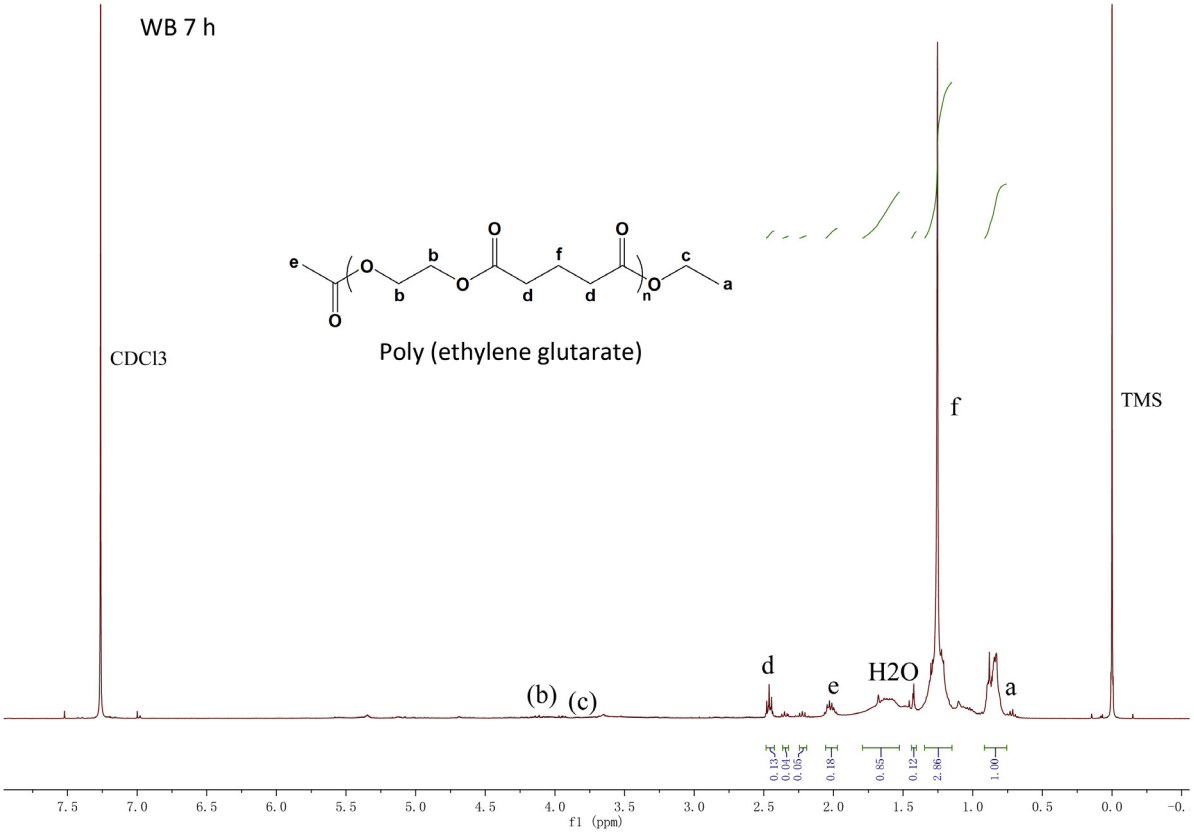


Fig. 2. H NMR spectrum of cotton fabrics Soxhlet's extraction liquids in $CDCl_3$. Cotton fabrics treatment reaction was performed in water bath (WB) for 7 h at 40 °C with an enzyme loading of 25% (v/v). Letters a, b, c, d and e correspond to C—H bonds.

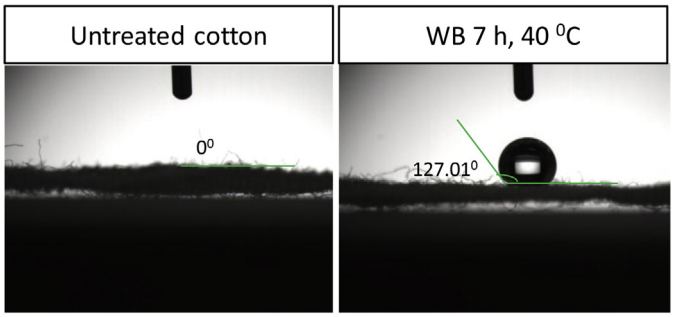


Fig. 3. Contact angles of treated and untreated cotton fabrics performed at room temperature with ultrapure water as test liquid. Cotton fabrics treatment reaction was performed in water bath (WB) for 7 h at 40 °C with an enzyme loading of 25% (v/v).

bicity improvement for cotton cellulose is critical for potential functional applications including rainwear, stain resistant products and water repellent outdoors gear [22,25]. As shown in Fig. 3, the treated cotton fabric became hydrophobic after *in situ* modification with oligomers of poly (ethylene glutarate). The results of the cotton fabric incubated with the reagents and without lipase are not shown because they were similar to the results of the untreated cotton fabric control.

The fabric had a contact angle of 127.01° due to the coating of poly (ethylene glutarate) oligomers at the surface of cotton fabrics after enzymatic treatment while the untreated cotton fabric had a contact angle of 0°. The increase on the contact angle of the treated cotton fabric after *in situ* modification with oligomers of poly (ethylene glutarate), was directed related with an improvement of the hydrophobic character of the cotton fabric. The existence of numerous ester groups in oligomers entrapped in the cotton contributed to the water repellence of the cotton fabrics.

Though the poly (ethylene glutarate) oligomers were not linked to the glycoside units of cotton cellulose, they were entrapped/networked on the surface cotton fibers. After washing the samples with chloroform, which removes the untrapped poly (ethylene glutarate) monomers and oligomers, the cotton fabrics remained hydrophobic (Fig. 3). Here we were aiming to make a prove of concept of coating and leaching experiments should be done in a next study. We would expect the oligomers would stay during washing and would be particialy remove by friction. The hydrophobic nature would keep the oligomers at the surface of fibre during washing and non-linkage would make the oligomers removable by friction, in similar fashion to the fastness of vat and reactive dyes.

Further, a water solution with bromophenol blue was used to study the significant water-repellence performance of the modified cotton (Fig. 4). The bromophenol blue droplets could keep on the surface of the treated cotton fabrics without almost any absorption even at the end of 32 s. This solid visual evidence supported the hydrophobic modification of cotton fabrics with poly (ethylene glutarate).

3.3. *Wrinkle recovery analysis of cotton fabrics*

Crease or wrinkle recovery was defined as the ability of a fabric to recover from the folding deformations [28]. This property of cotton fabric was considered as one of the essential performance parameters for evaluating the fabric wearability [31] and [32]. The wrinkle recovery angles of the treated cotton fabrics and the untreated cotton were recorded (Fig. 5). The crease recovery property was improved using the *in situ* enzymatic formation of

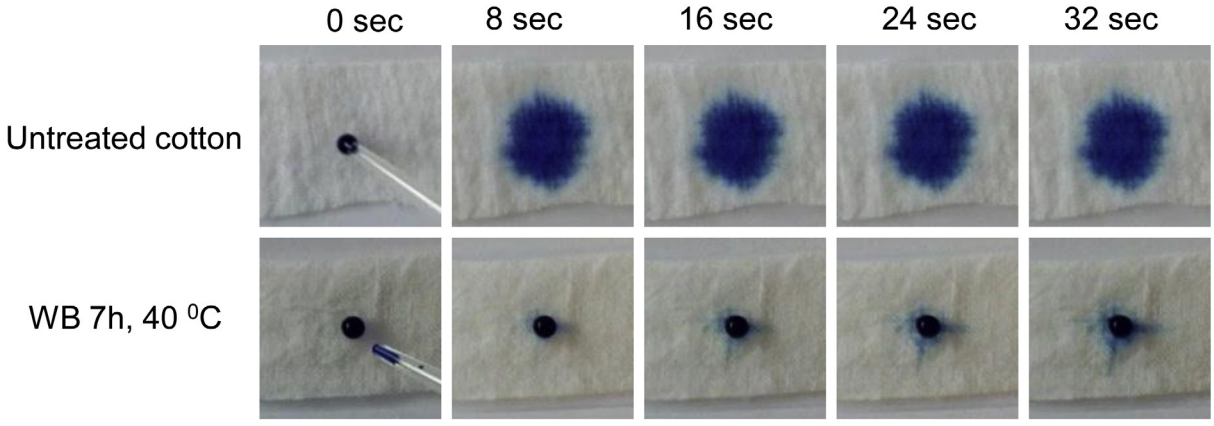


Fig. 4. Comparison of the absorption profiles of a droplet of bromophenol blue solution in cotton fabric treated by a lipase-catalyzed polyesterification reaction in water bath (WB) for 7 h at 40 °C and in the untreated cotton fabric.

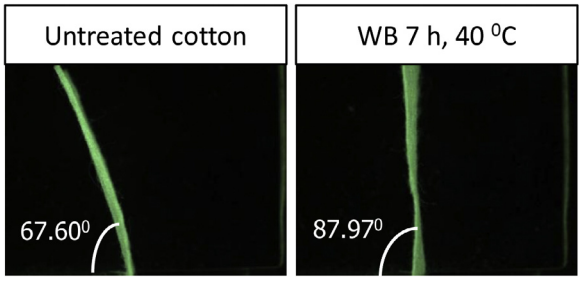


Fig. 5. Wrinkle recovery angles of the cotton fabric treated by a lipase-catalyzed polyesterification reaction in water bath (WB) for 7 h at 40 °C and the untreated cotton fabric.

poly (ethylene glutarate) monomers and oligomers at the surface of cotton fabrics. The wrinkle recovery angles of cotton fabric treated in water bath for 7 h was increased by 30.13% basing on that of untreated cotton.

4. Conclusion

Lipase-catalyzed solvent-free synthesis of poly (ethylene glutarate) monomers and oligomers to coat the surface of cotton fabrics was successfully carried out for the first time. A wider range of oligomers were found by MALDI-TOF mass spectra analysis with an overall avarage value of 2 (NMR data). The treated cotton had a hydrophobic behavior with a contact angle of 127.01° and an increase of wrinkle recovery of 30.13%. The *in situ* enzymatic formation of poly (ethylene glutarate) monomers and oligomers on the cotton fabrics resulted in coated fabrics with improved properties like wrinkle recovery and hydrophobicity. In future other reaction conditions will be tested in other to increase the properties and performance of the modified cotton fabrics.

The present work shows the potential of solvent-free lipase-driven reactions on the modification of the surface of cotton fabrics which can result in a wide range of applications on the textile industry.

Acknowledgements

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