

Antibacterial and Wrinkle Resistance Improvement of Nettle Biofiber Using Chitosan and BTCA

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Abstract

In this study, the possible improvement of the antibacterial and wrinkle resistance performance of 100% nettle fabrics was investigated. To realise this aim, antibacterial and wrinkle resistance finishing processes were applied. 1,2,3,4-butanetetracarboxylic acid (BTCA) and sodium hypophosphite (SHP) were used to impart the wrinkle resistance property. Moreover chitosan was incorporated in the finishing bath for the antibacterial property. The effects of respective treatments on the physical properties were determined and compared along with their antibacterial activity. BTCA concentration in the solutions influenced the physical properties of the nettle fabrics and 6% BTCA usage was found to be the optimum concentration rate. The addition of BTCA to the chitosan caused an improvement in the wrinkle resistance and slightly softer handle, in comparison with pure chitosan treatment; however, the strength loss slightly increased, as expected. The FTIR-ATR spectra showed a new peak that confirmed the ester linkage formation and crosslinking reaction.

Key words: antibacterial, biofiber, BTCA, chitosan, nettle, wrinkle resistance.

many, Austria, Great Britain, Finland and France prior to the introduction of cotton [4-6]. Since it had a biodegradable structure and could be produced easily from renewable sources, nettle fiber was used both for textile purposes and for cosmetics and medicine [4, 6, 7]. Moreover Bodros & Balely suggested that nettle biofibers can be used as reinforcing materials in composite structures due to their tensile properties [4].

Nettle biofiber displays good fiber properties such as length, fineness, strength as well as antistatic and UV transmittance for textile purposes and textile processing. The superb moisture absorption property and breathability of nettle fiber lead to better comfort properties for the textile end-use product. Nettle biofiber usage provides bio-degradable, renewable, sustainable and eco-friendly textile production, and has low energy consumption during production [4]. Recent studies have displayed that cotton fibers could be replaced by nettle fibers in many different applications owing to nice and suitable textile performance [7]. As a textile material, it has been used in the production of rope, string, cloth, tents, rucksacks, shirts, T-shirts, undershirts, upper garments, jackets, denim fabrics, socks, tablelinens, upholstery, carpets, bedding and camouflage equipment [4, 6, 8, 9].

As in other cellulosic bast fibers such as flax and hemp, nettle fiber fabrics can experience an undesirable wrinkling problem during their usage. Indeed wrinkling is an important issue in cellulosic fabrics

and it is mainly caused under a distorting force and moist conditions by the rupture and reformation of hydrogen bonds that hold the cellulose chains together [10-14]. To impart wrinkle resistance to cellulosic fiber based fabrics, crosslinking should be provided, and for this aim various crosslinking chemical agents have been tested up to now [12, 15-19]. However, most of them were eliminated because of ecological, economical and deteriorative reasons or due to their ineffectiveness. Today *N*-methylol reagents and multifunctional carboxylic acids are generally used [13, 14, 19].

Since the determination of formaldehyde as a probable human carcinogen chemical material, formaldehyde-free crosslinking agents like polycarboxylic acids have become more important than traditional *N*-methylol reagents [10-12, 15, 16, 19-26]. From the extensive studies on polycarboxylic acids, BTCA (1, 2, 3, 4-butanetetracarboxylic acid) was found to be the most effective crosslinking agent [11, 12, 16, 20-23, 27-29].

The crosslinking mechanism of polycarboxylic acids is based on the esterification of cellulose chains through the formation of an intermediate cyclic anhydride of polycarboxylic acid and its reaction with the hydroxyl groups of cellulose [11, 12, 18, 25]. The esterification reaction can be accelerated with the use of a proper catalyst, with sodium hypophosphite (NaH_2PO_2) known to be the best, especially for BTCA [11, 12, 28, 29]. In the application, sodium hypophosphite first-

Introduction

Bio-fibers are purely derived from vegetal sources that are fully biodegradable in nature, finding many different applications throughout the biomedical, food packaging, geotextile, architecture, composites and automotive transportation fields [1]. Indeed agro-based biofibers possess a composition, properties and structure that make them fit for uses such as pulp, paper, composites, and textile manufacture, and they can also be used for producing fuel, chemicals, enzymes as well as food [2]. Nettle fiber is one of the important biofibers [3]. The nettle plant belongs to the *Urticaceae* family and contains high quality cellulosic fibers like other bast fibers, such as flax and hemp. Nettle biofiber (*Urtica dioica* L.) was first used as a textile plant in Ger-

ly reacts with the intermediate cyclic anhydride of polycarboxylic acid and then with the hydroxyl groups of cellulose [29]. However, in earlier studies, it was determined that despite the fact that BTCA and SHP treatment had good results in terms of the wrinkle recovery angle, it has negative effects on the tensile and tear strength as well as on the colour properties of cellulosic fabrics [11, 17, 22]. This fact was attributed to the fiber degradation caused by the acid catalyst at high curing temperatures and by the limitation of stress distribution within the fibers due to their rigid crosslinking [15, 16, 23]. To minimise these side effects, a biopolymer addition to the finishing bath could be feasible.

It is known that chitosan [β -(1-4)-2-amino-2-deoxy-D-glucopyranose] biopolymer, which is obtained through chitin deacetylation, exhibits antibacterial properties due to its cationic amino groups [12, 17, 28, 30-38]. Cationic amino groups hold the negatively charged cell walls of microorganisms and inhibit their growth [30, 33, 34, 37, 39]. Moreover chitosan biomaterial is also biocompatible, biodegradable and non-toxic [28, 31-37], and has a film-forming property [38]. Chitosan biopolymer can be used in the textile industry for many purposes like chitosan fibers, wool pretreatment, dyeability improvement and antibacterial finishing, and it can form crosslinks with cellulosic fibers with the help of its cationic nature [16, 30, 33, 36, 38]. Hebeish *et al.* applied a multifinishing formulation containing BTCA and chitosan to cotton fabrics by the pad-dry-cure method [35]. They found that this treatment provided high crease recovery and antimicrobial activity with acceptable strength losses. Sauperl&Volmajer-Valh used the chitosan/BTCA combination system on viscose fabrics and concluded that this system provided good efficiency in terms of the breaking strength, wrinkle recovery angle and antimicrobial activity [40]. Bhuiyan *et al.* applied chitosan to jute fabric in order to improve the absorption of reactive dye and found that it was effective [36]. However, its usage in the textile industry is limited due to its weak binding and insufficient wash fastness [30, 31]. To overcome this problem, various derivatives of chitosan have been synthesised or various crosslinking agents have been tried [28, 30, 32, 39, 41, 42]. For instance, Kim *et al.* used a water soluble chitosan quaternary ammonium derivative to give antimicrobial activity

Table 1. Composition of finishing baths. **Note:** *BTCA: 1,2,3,4-butanetetracarboxylic acid and its catalyst SHP: sodium hypophosphite.

Treatments*	Chitosan, g	Acetic acid, ml	BTCA, g	SHP, g	Pure water, ml
1% Chitosan	3	6	–	–	294
4% BTCA + 4% SHP	–	–	12	12	300
6% BTCA + 4% SHP	–	–	18	12	300
8% BTCA + 4% SHP	–	–	24	12	300
12% BTCA + 4% SHP	–	–	36	12	300
1% Chitosan + 6% BTCA + 4% SHP	3	6	18	12	294

to cotton fabrics and to improve the laundering durability, in which they applied DMDHEU, BTCA and CA (citric acid) as crosslinkers [39]. They found that the cotton treated with HTCC and BTCA exhibited the best results. Montazer & Afjeh prepared quaternary ammonium salt of chitosan and combined it with crosslinking agents to obtain fabric with durable antimicrobial and crease free properties [32]. Cheng *et al.* derived modified chitosan and they coated it onto cotton fabrics with the help of BTCA [42]. It was found that this treatment improved crease recovery, but it resulted in damage to the breaking strength and bending properties of the fabrics.

The purpose of this study was to improve antibacterial and wrinkle resistance properties of the nettle biofiber fabrics using chitosan biopolymer, BTCA and SHP combination and finally to evaluate the effects of these treatments on the physical properties of the nettle fabrics.

Experimental

Materials

As a material, 100% plain woven nettle biofiber fabric with a weight of 295 g/m² was supplied by Octans Fabrics, South Korea. In the experiments, medium molecular weight chitosan biopolymer, acetic acid, 1,2,3,4-butanetetracarboxylic acid (BTCA) and sodium hypophosphite (SHP) supplied from Sigma Aldrich (USA) were used.

Methods

Antibacterial and wrinkle resistance finishing treatments

Chitosan (1% w/v) was dissolved in an acetic acid solution with a concentration of 2% (v/v). BTCA (4, 6, 8 and 12% conc.) and SHP (4% conc.) were prepared and applied to the nettle fabrics. In addition to these, a combination solution containing 1% chitosan, 6% BTCA and 4% SHP was also tested. The concentrations were calculated based on the bath

weight, and the compositions of the finishing baths are given in **Table 1**.

Finishing agents (chitosan, BTCA and SHP) were applied to the nettle fabrics by the pad-dry-cure method. The nettle biofiber fabrics were immersed in aqueous solutions containing chemicals in the aforementioned concentrations for 5 minutes and then padded through squeeze rolls to give a 90% wet pick-up with two dips and two nips. After that, the nettle fabrics treated were dried at 85 °C for 5 minutes and then cured at 170 °C for 3 minutes.

Wrinkle recovery angle (WRA) measurement

The WRA [Wrinkle Recovery Angle, °] of the nettle samples was measured for both warp and weft directions according to the TS 390 EN 22313 standard with an SDL Atlas Wrinkle Recovery Tester (USA).

Tensile strength measurement

The tensile strength and elongation at break values of the nettle fabric samples were measured according to the TS EN ISO 13934-1 standard using a Tinius Olsen Strength Testing Machine (USA).

Bending measurement

The bending length and bending rigidity values of the nettle fabric samples were measured according to the TS 1409 standard using a Shirley Stiffness Tester (USA). Bending rigidity values were calculated according to **Equation (1)**.

$$G = 0.1 \times W \times L^3 \text{ (mg} \times \text{cm)} \quad (1)$$

G: bending rigidity, W: weight of the fabric, 295 g/m², L: bending length, cm

Antibacterial activity assessment

Antibacterial assessment against *Staphylococcus aureus* (ATCC 29213, gram-positive bacteria) and *Escherichia coli* (ATCC 25922, gram-negative bacteria) was carried out quantitatively according to the Shake flask method

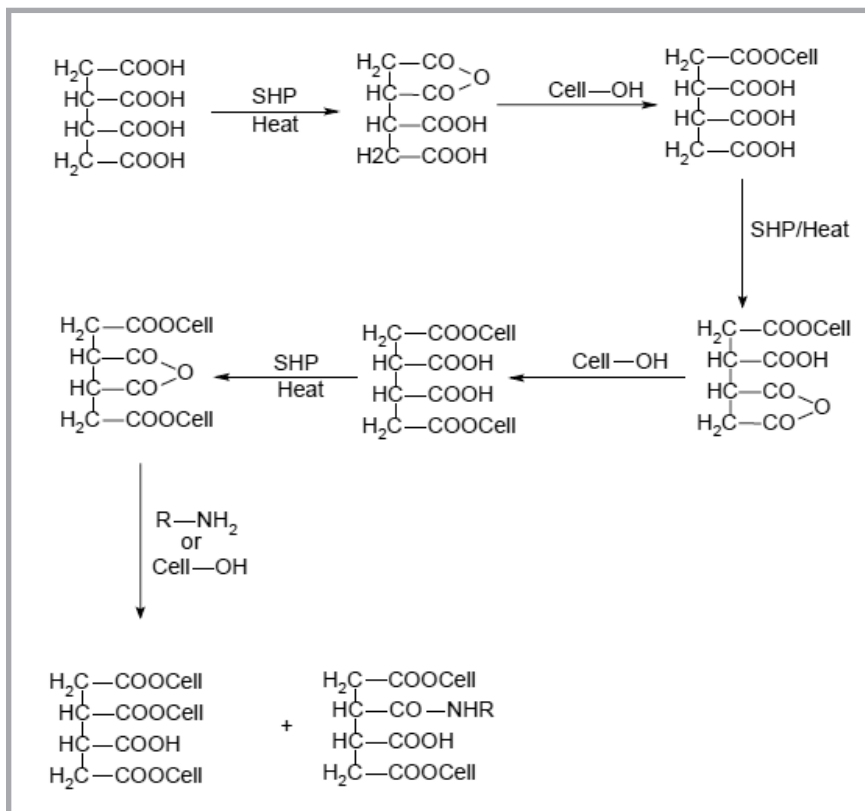


Figure 1. Crosslinking mechanism of BTCA on cotton cellulosic fiber with the presence of chitosan biopolymer [28].

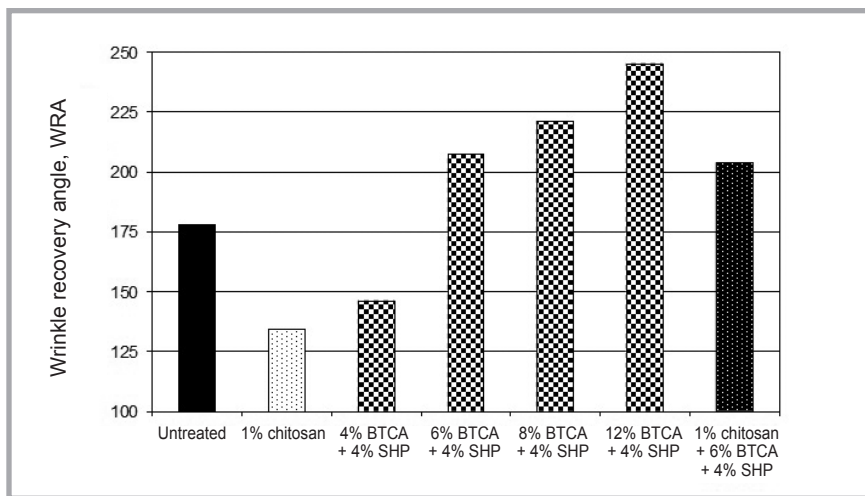


Figure 2. Total WRA angles, ° of treated and untreated nettle fabric samples.

Table 2. Wrinkle recovery angle (WRA) values of the treated and untreated nettle fabric samples. **Note:** *BTCA: 1,2,3,4-butane-tetracarboxylic acid and its catalyst SHP: sodium hypophosphite.

Samples*	Warp WRA, °	Weft WRA, °
Untreated nettle	74	104
1% chitosan treated nettle	58	76
4% BTCA + 4% SHP treated nettle	66	80
6% BTCA + 4% SHP treated nettle	91	116
8% BTCA + 4% SHP treated nettle	97	124
12% BTCA + 4% SHP treated nettle	118	127
1% chitosan + 6% BTCA + 4% SHP treated nettle	89	115

(ASTM E2149-01). The decrease in the number of living micro-organisms was calculated from the number of living micro-organisms present in the medium containing the sample according to the following **Equation (2)**:

$$\text{Reduction (\%)} = [(A-B)/A] \times 100 \quad (2)$$

Where, A is the number of living micro-organisms (CFU/ml, colony forming units per milliliter) before shaking; and B is the number of living microorganisms (CFU/ml) after shaking.

The antibacterial test was applied only to two samples. Only the untreated sample (as a control) and chitosan-BTCA-SHP combination treated sample, which is representative of samples exhibiting good easy-care properties, were tested for the anti-bacterial property. It is already known that chitosan gives anti-bacterial activity, therefore only the chitosan treated sample was not tested here. However, herein our main topic was to examine whether both the easy-care and antibacterial properties could be attained on nettle fiber fabric at the same time.

FTIR spectroscopy

Infrared analysis was performed using a Perkin Elmer (USA) Spectrum Two™ Infrared Spectrometer (FT-IR) with the diamond universal ATR accessory in the ATR mode, which employs a Diamond crystal giving an effective depth of penetration of 1 micron at a resolution of 4 cm⁻¹. The spectrum recorded for each sample was the average of 4 scans.

Results and discussion

The crosslinking mechanism of BTCA in the presence of chitosan biopolymer in the cotton fabrics is shown in **Figure 1**. Since the nettle biofibers are also of cellulosic origin, this mechanism can be associated with nettle fibers as well.

Wrinkle recovery angle (WRA) evaluation

WRA (wrinkle recovery angle) values of the treated and untreated nettle biofiber fabrics are exhibited in **Table 2**. In addition to warp and weft values, the total WRA angles, ° are also reported in **Figure 2**.

An increment on the wrinkle recovery angle exhibits a decrease in the fabrics' crease tendency, where they can easily

turn to their original initial state before folding, and do not crease [43].

As a result of the measurements, it was observed that some treatments resulted in a decrease in the wrinkle recovery angle, whereas some of them led to an increase in the WRA (*Table 1* and *Figure 2*). For instance, pure chitosan biopolymer treatment and 4% BTCA + 4% SHP treatment decreased the WRA, but on the other hand, when the BTCA rate was higher than 6%, significant increases were observed. This fact showed that crosslinking of the cellulosic hydroxyls began at this concentration rate. Since the use of 6% BTCA concentration along with 4% SHP affected the tensile strength less than the other concentration rates studied, as well as providing an increase in the wrinkle recovery angle (*Tables 1* and *2*), the acceptable BTCA rate was determined as 6% for the combination study. Combination treatment was applied as 1% chitosan + 6% BTCA + 4% SHP. In this treatment, WRA values were found to be 89° and 115° in the warp and weft directions, respectively (*Table 1*). In addition, it was also observed that there was about a 15% increase in the total WRA in the case of 1% chitosan + 6% BTCA + 4% SHP combination treatment (*Figure 2*). This enhancement was attributed to the crosslinking between BTCA and hydroxyl groups of nettle cellulose and the formation of hydrogen bonds by means of chitosan, BTCA and SHP. This fact was also earlier reported in previous studies about other cellulosic fibers [11, 28, 35].

Tensile strength evaluation

Tensile strength, tensile strength loss, % and elongation at break, % values of the nettle biofiber fabrics treated are given in *Table 3*.

According to the measurement values, it was observed that the tensile strength and elongation at break values of the samples treated were lower than those of the untreated nettle sample. The treatment that affects the tensile strength and elongation at break values at the minimum rate was found to be pure chitosan treatment. As the BTCA concentration rate in the solutions increased, the loss in tensile strength became distinctive, and when a 12% BTCA concentration rate was applied, the strength loss reached 71% (*Table 2*). On the other hand, the combination treatment (1% chitosan + 6% BTCA + 4% SHP) resulted in a 48.5% strength loss (*Table 2*). The loss in the tensile

Table 3. Tensile strength properties of treated and untreated nettle fabrics. *Note:* *BTCA: 1,2,3,4-butanetetracarboxylic acid and its catalyst SHP: sodium hypophosphite.

Treatment types*	Total wrinkle recovery angle, °	Tensile strength, N	Tensile strength loss, %	Elongation at break, %
Untreated	178	1156	–	15.14
1% chitosan	134	695	40.00	14.11
4% BTCA + 4% SHP	146	622	46.00	11.85
6% BTCA + 4% SHP	207	540	53.00	11.05
8% BTCA + 4% SHP	221	434	62.50	8.62
12% BTCA + 4% SHP	245	338	71.00	7.46
1% chitosan + 6% BTCA + 4% SHP	204	596	48.50	11.78

Table 4. Bending length and bending rigidity values of treated and untreated nettle fabrics. *Note:* *BTCA: 1,2,3,4-butanetetracarboxylic acid and its catalyst SHP: sodium hypophosphite.

Treatment type*	Total wrinkle recovery angle, °	Tensile strength loss, %	Bending length, cm	Bending rigidity, mg x cm
Untreated	178	–	4.92	3513.32
1% chitosan	134	40.00	9.60	26099.7
4% BTCA + 4% SHP	146	46.00	6.07	6597.63
6% BTCA + 4% SHP	207	53.00	6.57	8366.01
8% BTCA + 4% SHP	221	62.50	6.60	8481.13
12% BTCA + 4% SHP	245	71.00	6.95	9903.22
1% chitosan + 6% BTCA + 4% SHP	204	48.50	9.40	24502.2

strength property was expected and was also observed in former studies about other cellulosic fabrics, being attributed to the depolymerisation of cellulose macromolecules depending on the acidity of the treatments as well as on the oxidation and scission of cellulosic chains during the fixation process and rigid crosslinking between fibers [11, 16, 18, 23, 28, 40, 42]. When the elongation break values are considered, it can be said that the differences between samples observed were negligible and the elongation decrease observed can be associated with the crosslinking increase.

Bending length and bending rigidity evaluation

Bending length and bending rigidity values of the nettle biofiber fabrics treated are displayed in *Table 4*.

The higher bending length and bending rigidity values mean that the fabric becomes stiffer and exhibit higher resistance to bending [42, 43]. It can be easily seen from the bending length and bending rigidity results of *Table 3* that the treatment that stiffens the fabric handle the most was found to be pure chitosan application. Or in other words, chitosan treatment alone led to the stiffest handle in this study (*Table 3*), likely arising from

the surface coating effect of the chitosan biopolymer [28]. But when chitosan biopolymer was combined with BTCA (1% chitosan + 6% BTCA + 4% SHP), the bending length and bending rigidity values decreased, and therefore a little improvement was observed. Moreover BTCA treatments also affected the fabric handle negatively, but not as much as the pure chitosan application, and it can be seen that the higher the BTCA rate in the solutions, the stiffer the fabric handle.

When the results were considered generally, it was understood that 1% chitosan + 6% BTCA + 4% SHP combination treatment was feasible not only to minimise the loss in physical properties but also to obtain antibacterial and wrinkle resistant nettle fabric.

Antibacterial activity evaluation

Antibacterial activity results of the nettle fiber fabric samples are shown in *Table 5*.

According to the test results, it was found that the chitosan-BTCA-SHP combination treated nettle biofiber fabric sample exhibited good antibacterial activity against *S.aureus* and *E.coli*, which was in line with the earlier findings of previous studies but for different fibers [17, 28, 32, 35]. In addition, it can be said that the

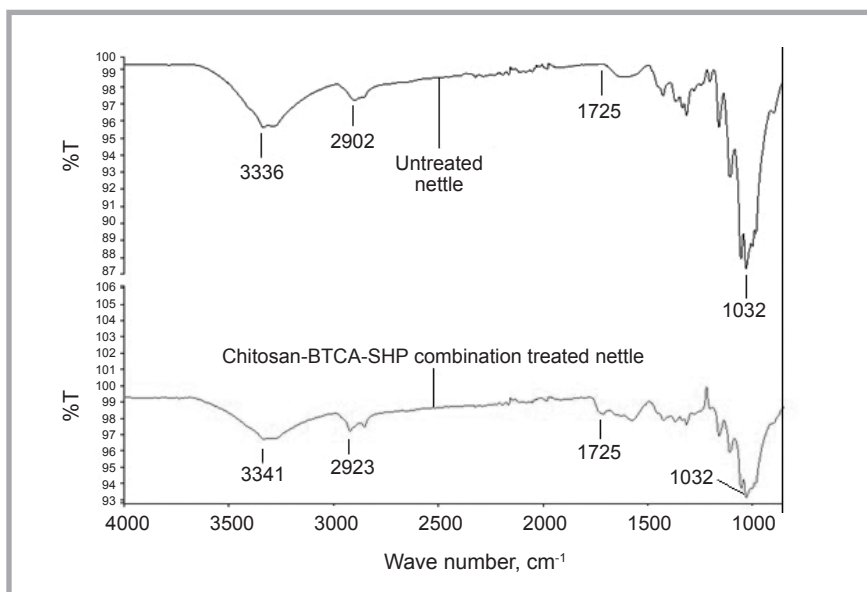


Figure 3. FTIR-ATR spectra of the untreated nettle and chitosan-BTCA-SHP combination treated nettle biofiber fabrics

effect of the combination treatment (1% chitosan + 6% BTCA + 4% SHP) was higher against gram positive than gram negative bacteria (Table 4). This fact was closely related to the difference in the chemical structure of the bacteria, and similar observations were also reported in other studies related to other fibers [28, 42]. Although the untreated nettle biofiber fabric sample exhibited some antibacterial activity against *S.aureus* as well, this antibacterial activity was not high enough.

Infrared spectroscopy (FTIR-ATR) analysis

Figure 3 shows FTIR-ATR spectra of the untreated nettle biofiber fabric sample and chitosan-BTCA-SHP combination treated nettle sample. As seen in the spectra, at 1725 cm⁻¹ the treated sample showed an ester band confirming the formation of a crosslinking reaction, unlike the untreated sample. It was also observed that the cellulosic bands of 3336 and 2902 cm⁻¹ shifted to 3341 and 2923 cm⁻¹, respectively, and the absorb-

ance intensity of the band at 1032 cm⁻¹ decreased after treatment. This could be attributed to the conversion of carboxyl groups into ester linkages and decrease in the total numbers of carboxyl groups due to the crosslinking. These results were in line with the findings of former studies on cotton, viscose, flax and ramie fibers [18, 21, 23, 26, 44, 45].

Conclusion

The aim of this research was to improve the antibacterial and wrinkle resistance properties of nettle biofiber fabrics using chitosan biopolymer, 1,2,3,4-Butanetetracarboxylic acid (BTCA) and its catalyst – the sodium hypophosphite (SHP) combination. BTCA concentration in the solutions influenced the physical properties of the nettle biofiber fabrics, and 6% BTCA usage was found to be the most suitable and optimum concentration rate. On the other hand, 1% pure chitosan biopolymer application treatment alone resulted in a negative effect on the wrinkle property, with a lower recovery angle

and higher stiffness with the higher bending length value leading to a more creasable and stiffer handle in comparison to untreated nettle biofiber fabric.

In order to achieve an improvement in both wrinkle resistance and antibacterial activity, chitosan and BTCA along with its catalyst – SHP were applied to nettle biofiber fabric as a combination treatment. The addition of BTCA to the chitosan treatment caused an improvement in wrinkle resistance and slightly softer handle, in comparison with chitosan treatment alone; however, the strength loss slightly increased, as expected. Indeed the chitosan and BTCA combination process (1% chitosan + 6% BTCA + 4% SHP) provided satisfactory results with regard to wrinkle resistance and antibacterial finishing for nettle fabrics. The combination process provided a higher reduction rate of bacteria as compared to the untreated nettle fabric. Moreover FTIR-ATR spectra of the combination process treated nettle sample showed a new peak that confirmed ester linkage formation and crosslinking reaction. The combination treatment (1% chitosan + 6% BTCA+4% SHP) resulted in 15% wrinkle resistance improvement, measured by the total wrinkle recovery angle, and stiffer handle, measured by the bending length, higher antibacterial activity and expected strength loss when compared with untreated nettle biofiber fabric.

Overall the nettle biofiber fabric finished with 1% chitosan + 6% BTCA + 4% SHP combination treatment displayed adequate wrinkle resistance, tensile strength and handle properties along with improved antibacterial activity.

Analysis of the finishing resistance in the conservation process was not conducted in detail here, since this was a preliminary study. This will be the scope of our next study, from a different point of view and with new chemical combinations.

Table 5. Antibacterial activity results of the untreated and chitosan-BTCA-SHP combination treated nettle fabric samples (bacterial reduction rate, %). Note: *BTCA: 1,2,3,4-butanetetracarboxylic acid and its catalyst SHP: sodium hypophosphite; **The bacterial reduction rate, which is less than 10%, is accepted as no antibacterial activity.

Treatment types*	Total wrinkle recovery angle, °	Antibacterial activity (bacterial reduction rate, %)	
		<i>Staphylococcus aureus</i>	<i>Escherichia coli</i>
Untreated	178	32.80	– **
1% chitosan + 6% BTCA + 4% SHP	204	99.99	56.89

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