The influence of biopolymer coatings on the physical-mechanical properties of yarns

Utjecaj biopolimernih prevlaka na fizikalno-mehanička svojstva pređe

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Abstract

The modification of polyester yarn was carried out with the aim of producing sustainable coatings from from erythritol, gelatine and collagen with the addition of propolis and alginate fibres and a natural plasticiser (glycerine). The advantage of the sustainable coatings is that they consist of compounds that can be taken orally and are therefore suitable for external use. The morphology of the yarn was determined before and after modification with Dino-Lite. The pH value, thickness and linear density were also monitored before and after modification. The results showed that the modification had no significant effect on the thickness or linear density of the yarns. Furthermore, based on the pH results, the modified samples are suitable for external application on the skin. The results of the tensile strength properties of the modified samples showed a decrease in the strength value of the tested samples (the untreated sample has the highest value and the sample treated with erythritol and propolis has the lowest). When analysing the elongation values of the tested samples, it is noticeable that the sample treated with erythritol and propolis has the lowest ability after modification, while the untreated sample also has the highest elongation ability. The modified samples had a very pleasant hand value. The modification of polyester yarn was carried out with the aim of obtaining a future flat textile product that can be used as a medical dressing for chronic wounds.

Keywords: medical textile; yarn; modification; testing; biodegradable natural polymers

Sažetak

U ovom radu provedena je modifikacija poliesterske pređe, s ciljem razvoja održivih prevlaka od eritritola, želatine i kolagena uz dodatak propolisa i alginatnih vlakana te prirodnog plastifikatora (glicerina). Prednost održivih prevlaka je što se sastoje od spojeva koji se mogu uzimati oralno i stoga su prikladni za modifikaciju. Morfologija pređe određena je prije i nakon modifikacije pomoću Dino-Litea. Također su praćeni pH vrijednost, debljina i finoća pređe prije i nakon modifikacije. Rezultati su pokazali da modifikacija nije imala značajan učinak na debljinu ili finoću pređe. Nadalje, na temelju pH rezultata, modificirani uzorci su prikladni za vanjsku primjenu na koži. Rezultati svojstava vlačne čvrstoće modificiranih uzoraka pokazali su smanjenje vrijednosti čvrstoće ispitivanih uzoraka (neobrađeni uzorak ima najveću vrijednost, a uzorak obrađen eritritolom i propolisom ima najmanju). Pri analizi vrijednosti istezanja ispitivanih uzoraka uočava se da uzorak obrađen eritritolom i propolisom ima najmanju sposobnost istezanja nakon obrade, dok neobrađeni uzorak također ima najveću sposobnost istezanja. Modificirani uzorci bili su vrlo ugodnog opipa (engl. hand value). Modifikacija poliesterske pređe provedena je s ciljem dobivanja budućeg plošnog tekstilnog proizvoda koji se može koristiti kao medicinska obloga za kronične rane.

Ključne riječi: medicinski tekstil; pređa; izmjena; ispitivanje; biorazgradivi prirodni polimeri

1. Introduction

Biopolymers are polymers produced in nature or from natural raw materials and those produced from petroleum derivatives that may be biodegradable [1]. They represent a large group of biomaterials whose most important property is that they are biocompatible, non-toxic and non-cytotoxic and that they yield degradation products that are themselves non-toxic. Biodegradable polymers are, in a broader sense, those polymers that degrade in the biological environment: in the soil, in the sea, in rivers, in lakes, in the human or animal body through enzymatic or non-enzymatic hydrolysis [2]. In a narrower sense, biodegradation is decomposition that only occurs through the enzymatic action of microorganisms, fungi or bacteria [3]. The advantages of using such polymers are the reduction of greenhouse gas emissions into the atmosphere and the reduction of environmental pollution. The raw materials used for their production come from renewable sources, their waste biomass can also serve as a raw material, and they have similar properties to synthetic ones. The use of biodegradable polymer materials is expected to reduce the need to produce synthetic polymers (and therefore pollution) and will have a positive effect from both an environmental and economic perspective. In view of this, the modification of textiles with biodegradable polymers is certainly an alternative to the usual textile treatments, in particular the sol-gel process [4]. The sol-gel process, like many other processes in the textile industry, essentially

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stands for the modification of textiles with synthetic compounds, while the modification of textiles with biopolymers represents a switch to sustainable, biodegradable and natural polymers. Natural polymers have a much more complex structure than synthetic polymers, which have a relatively simple structure. This complexity of natural polymers offers a number of advantages in the modification of textiles with the aim of obtaining potentially biocompatible, non-toxic and non-cytotoxic medical or cosmetic materials (wound dressings, cosmetic wipes/pads, yarns as potential surgical threads, etc.).

The most important representatives of biopolymers derived from biomass are polysaccharides and polypeptides (proteins). Polysaccharides include thermoplastic starch, cellulose and its derivatives, fibres, chitin and chitosan, rubber. The group of polypeptides (proteins) includes wheat gluten, soya protein, collagen and gelatine as well as whey protein - casein.

Collagen plays an important role in all phases of wound healing, as it attracts fibroblasts to the wound site due to its chemotactic properties, promotes the distribution of platelets at the site of injury and preserves leukocytes and macrophages [5]. It also supports the formation of new blood vessels, the formation of granulation tissue, the debridement of the wound and the ability of the wound to re-epithelialize [6].

Erythritol belongs to the group of sugar alcohols, also known as polyols, and is currently found alone or in combination with other polyols in foods, cosmetics and pharmaceuticals. Erythritol can suppress the growth of bacteria in the mouth as it has antibacterial potential against certain pathogens [7] and has been found to have a positive effect on the prevention of gingivitis by preventing the maturation of oral biofilms [8].

Gelatine is a colourless protein that is purified and extracted from a connective tissue called collagen, which is found in animal bones and skins [9]. Gelatine is a natural biopolymer that is naturally biocompatible and biodegradable, has low immunogenicity and is classified as "Generally Recognised as Safe" (GRAS) by the United States Food and Drug Administration (FDA) [10]. In the manufacture of capsules or tablets or as a component of wound dressings, haemostatic sponges or blood volume substitutes is used.

Glycerol (also known as glycerin) is widely used in medicine (allergen immunotherapies, cough syrups, elixirs and expectorants, etc.), in the cosmetics industry (creams, soaps, etc.), in pharmacy (as a binder for tablets, in medicines for burns, bites, cuts, psoriasis, etc.), in oral hygiene products (toothpaste, mouthwash, etc.) as an emollient, lubricant and humectant [11]. In this work, glycerol was used as an emollient and distilled water as a solvent. It was hypothesised that it can contribute to the development of sustainable coatings as a potential equivalent active ingredient in wound dressings (it retains moisture; wounds heal faster in a moist medium and meets pharmacopoeia requirements).
The Food and Drugs Administration (FDA) approved alginate as "Generally Recognised as Safe" (GRAS) for food, pharmaceutical and medical applications (wounds, bone) [12]. Alginate is widely used in biomedical research due to its versatile and biological properties such as biocompatibility, (possible) non-immunogenicity, chelating agent, water solubility, flexibility (it can be easily modified into any shape) and low cost [13]. In particular, alginate is used in protein/ drug delivery systems, tissue regeneration and wound healing [14].

Propolis is a natural resinous material produced by honeybees with a mixture of saliva and beeswax as well as substances from various parts of plants such as bark, buds and exudates [15, 16]. The chemical composition of propolis depends sufficiently on the characteristics of the local flora, honeybee species, climatic and geographical factors, collection times and plant resources [17]. A typical resinous mixture of propolis consists of 40%–70% balsam (phenolic acids and flavonoids), 20%–35% waxes, 1%–3% aromatic and essential oils and 5% other components such as vitamins, minerals, proteins and enzymes [18]. The wide use of propolis in modern medicine is mainly attributed to its phenolic acids and flavonoids, which have a broad spectrum of biological and pharmacological activities, including antioxidant, anti-inflammatory, hypoglycaemic, immunomodulatory, ant apoptotic, antifungal, antibacterial and anticancer properties [15].

In this work, natural, biodegradable polymers (erythritol, gelatin or collagen with propolis or alginate fibres) that are environmentally friendly and fulfil the requirements of the FDA and the EMA were used for the development of sustainable coatings.

2. Materials and Methods

2.1. Preparation of Materials and Sustainable Coatings

Commercially available yarn was used. The composition of the yarn was determined according to ISO/TR 11827:2012 and revealed that the yarn is polyester fibres; the yarn linear density was determined according to ISO 2060:2008 is 14 tex [19, 20]. Based on the properties of natural biodegradable polymers mentioned in the introduction and in our previous work [21, 22, 23], it was decided that sustainable coatings would be made from erythritol, gelatine, and collagen (Table 1). Numerous preliminary tests were carried out, were different ratios of the polymers to water as a solvent, duration of the preparation process, and temperature were selected.

The main idea of this work was to choose the best modification of erythritol, gelatine, and collagen, taking into account the requirements for maintaining the textile character, transparent and translucent sustainable coating on yarn. The best modification should used as a basis for the separate addition of propolis and alginate fibres. In this work, the glycerol was used as a plasticizer to prevent denaturation of the chains during the reaction.

Table 1. Properties of polymers.

Polymer	Molar mass [g mol^{-1}]	CAS number	Note
Erythritol $C_4H_{10}O_4$	122.12	149-32-6	from corn
Gelatin $C_6H_{12}O_6$	45.00	9000-70-8	100 % beef gelatin
Collagen $C_{57}H_{91}N_{19}O_{16}$	1302.5	/	fish collagen
Glycerol $C_3H_8O_3$	92.09	56-81-5	/
Propolis $C_{17}H_{16}O_4$	281.31	/	propolis extract in an aqueous solution with niacin and sage
Sodium Alginate Fibres	/	/	sodium alginate fibers with sodium and calcium carbonate

Each polymer (erythritol, gelatine, and collagen) has biocompatible and biodegradable properties, but the most important effects for sustainable coatings in this work, **erythritol** slows down bacterial growth in biofilms; **gelatin** has numerous functional groups that enable further surface functionalisation to achieve active targeting of diseased cells; **collagen** can create a moist healing environment that enables wound healing. The body's own collagen can be utilised for new tissue growth.

The modification of polymers with propolis or alginate fibres could represent a synergistic approach, **propolis** is a natural broad-spectrum antibiotic, and **alginate fibres** have the task of maintaining a physiologically moist microenvironment, minimising bacterial infection at the wound site.

Any modification on yarns could be part of a single-layer product or a multi-layer wound dressing against biofilms.

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Table 2. Schematic representation of the modifications carried out.

Erythritol / Gelatin / Collagen
+ distilled water
mixing on a magnetic stirrer
↓ ↓
+ propolis 10 ml
+ glycerol 5 ml
+ alginate 10 ml
+ glycerol 5 ml
20 ± 2 °C
15 min
↓
The samples were kept at 20 ± 2 for 24 h
↓
The samples were fixed at 40 °C for 60 min

At the beginning of the modification with erythritol, a solution of distilled water, erythritol and propolis was prepared (Table 2). The solution was mixed thoroughly. Glycerol was then added with increasing mixing speed. The process was carried out with constant magnetic stirring until a homogeneous solution was obtained. The yarns were then modified (coated) using the dip coating method on a dip coater at a predetermined drawing speed of 1 mm/s to obtain a thin coating. The modified samples were dried at room temperature for 24 hours and then at 40 °C for 60 minutes (Figure 1).

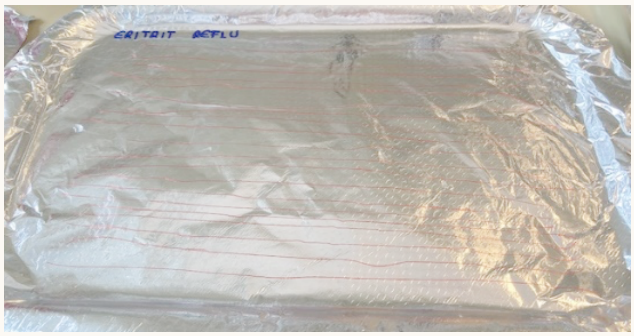


Figure 1. Samples dried at room temperature.

The experimental part of this work required the preparation of biodegradable natural polymers, i.e. the development of their optimal formulations, and then the application of physical-mechanical and other methods to characterise and test the resulting coatings on the yarn:

- Material morphology with Dino-Lite pH of water extract from wet-processed textiles
- Determination of thickness and mass per unit length
- Determination of one-sided breaking force and elongation at break with the CRE tester (Constant Rate of Extension)

2.2. Determination of the morphology of the yarns

The surface properties of the untreated and treated yarns were determined using the Dino-Lite Microscopy System Pro AM413T (Torrance, CA, USA). The digital microscope has a resolution of 1.3 megapixels and can achieve a magnification of up to 200x. The morphological structure of the material, the homogeneity or heterogeneity of the coatings, possible agglomerates of the coatings, thickened and thinned areas of the fibres and the thickness were observed.

2.3. Activity of the hydrogen ions

The hydrogen ion activity or pH of the water extract from wet-treated textiles was determined according to AATCC test method 81-1988 [24]. The sample was boiled in distilled water at 80 °C for 10 minutes. The water extract was then cooled to room temperature and the pH value was determined using a pH metre.

2.4. Determination of tensile properties

A standardised test of determination of breaking force, elongation and tenacity of yarns was used. The test was carried out in accordance with the following standard ISO 2062:2009 [25]. The test was performed on a constant elongation speed machine Tensolab 3000, Mesdan S.p.A., Italy.

3. Results and discussion

The tests in this work were carried out with a single linear textile – polyester yarn, which is commercially available. A yarn was selected in order to obtain as much relevant data as possible and to have the possibility to optimise as many parameters of the modification as possible. Table 3 shows the sample designations used in this work.

Table 3. Codes of the samples used in the work.

Code	Biopolymer	Active substance	Note
N	/	/	unmodified sample
EP	erythritol	propolis	/
EA		alginate	/
ZP	gelatin	propolis	/
ZA		alginate	/
KP	collagen	propolis	/
KA		alginate	/

3.1 Determination of the morphology of the yarns

The results of the determination of the yarn linear density (T_t) before and after modification are shown in Table 4.

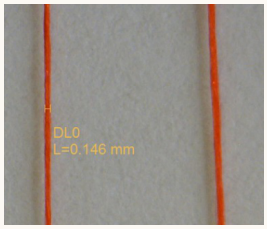
Table 4. Results of yarn linear density before and after modification.

Code	Tt_{before} [tex]	Tt_{after} [tex]
EP	14.00	14,7
EA	14.00	14.5
ZP	14.00	13.9
ZA	14.00	14.5
KP	14.00	14.4
KA	14.00	14.0

The linear density of the modified yarns is close to the values of the unmodified yarns. The samples modified with erythritol have the highest linear density (14.7 tex and 14.5 tex). Samples modified with gelatine have a linear density between 13.9 tex (with propolis) and 14.5 tex (with alginate fibres). The values of the modifications with collagen lie between the modifications with erythritol and gelatine. With the exception of one sample, the specific density of the substances used led to an increase in yarn thickness, which may be a result of the solidification of the samples and the coating remaining on the drying surface before weighing.

The yarn thickness was determined on samples before and after modification (Figure 2.) in such a way that 5 individual measurements were carried out. The results of the mean values are shown in Table 5.

a)



b)

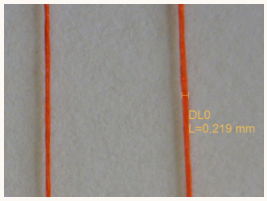


Figure 2. Thickness of yarn code EP (modification by erythritol and propolis) a) before modification, b) after modification.

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The results obtained show that the yarn thickness increases in all modified samples. The greatest increase in mass is observed in the sample modified with gelatine and alginate fibres (sample code ZA) and the least in the sample modified with collagen and propolis (sample code KP). The greatest increase in mass (sample modified with gelatine) can be interpreted by the gelling temperature of the gelatine, which is between 25 and 30 °C depending on the amount of dissolved gelatine. The phase transition is accompanied by a conformational change of the gelatine chains and the connection of three different gelatine molecules via triple helices to form a gel network. It is assumed that as the temperature is lowered, the gel network is increasingly connected to each other via individual bonds until the network permeates the entire sample and changes its aggregate state [26].

The collagen triple helix has certainly contributed to a better binding of the collagen to the yarn. Alginate fibres with sodium and calcium carbonate also contribute to gelation and thickness increase. The structure and properties of the gels formed depend on the chemical structure of the polysaccharide chain, i.e. the proportion of the individual uronic acids [27].

Table 5. Results of thickness (D) before and after modification.

Code	D_{before} [mm]	D_{after} [mm]	ΔD [mm]
EP	0.146	0.219	0.073
EA	0.255	0.292	0.037
ZP	0.146	0.219	0.073
ZA	0.109	0.365	0.256
KP	0.219	0.255	0.036
KA	0.146	0.255	0.109

3.2 Results of activity of the hydrogen ions

Unmodified and modified samples have a neutral pH value of (7) and are considered neither acidic nor alkaline. In this work, the achieved pH of the coating of 7 is considered a good basis for the addition of targeted substances to suppress the growth of certain microorganisms. Some studies have found that the pH environment of chronic wounds ranges from pH 7.15 to 8.9 and that the pH of the wound shifts towards an acidic value as healing progresses [28]. Most bacterial organisms grow best at pH values of 6.5 to 7.0; however, each microbial species has its own pH range in which it grows best.

3.3 Results of tensile properties

The results of the determination of the breaking force, the yarn elongation and the tenacity are shown in Table 6. The modification of the yarn with sustainable coatings of biodegradable natural polymers showed lower results in tensile properties than the unmodified sample. Modifications with erythritol showed the lowest tensile strength results, although the difference between modification with propolis (9.85 cN/tex) or alginate fibres (10.65 cN/tex) is negligible. The highest values were found in the modification with gelatine, where 13.38 cN/tex were achieved with propolis and 12.54 cN/tex with alginate fibres. Modifications with collagen showed similar values to gelatine, but a slightly higher value for the modification with propolis (13.52 cN/tex).

The modified samples showed low elongation values, indicating a low elongation potential. The unmodified sample has the highest values (9.78%) and the modification with erythritol and propolis the lowest (6.99%). The results for elongation show a similar behaviour to the results for tenacity.

Table 6. Results of single-end breaking strength, elongation of yarns and tenacity.

Code	Statistical parameter	F [cN]	ε [%]	R_s [cN/tex]
N	x	**2.0**	**9.78**	**14.75**
	σ	0.2	1.09	1.23
	V [%]	8.42	11.13	8.42
EP	X	**1.4**	**6.99**	**9.88**
	σ	0.2	1.58	1.761
	V [%]	17.83	22.53	17.83
EA	x	**1.5**	**7.89**	**10.65**
	σ	0.2	1.75	1.77
	V [%]	16.65	22.16	16.65
ZP	x	**1.9**	**8.88**	**13.38**
	σ	0.1	1.31	0.84
	V [%]	6.27	14.79	6.27
ZA	x	**1.8**	**8.52**	**12.54**
	σ	0.1	1.04	0.77
	V [%]	6.15	12.22	6.15
KP	x	**1.9**	**9.24**	**13.52**
	σ	0.3	2.01	1.87
	V [%]	13.83	21.79	13.83
KA	x	**1.8**	**8.52**	**12.54**
	σ	O.2	1.69	1.67
	V [%]	13.29	19.86	13.29

Where X is the arithmetic mean, σ is the standard deviation, V is the coefficient of variation and Rs is tenacity.

Note: The tests were performed on 50 samples for each modification.

4. Conclusion

In this work, natural, biodegradable polymers that are environmentally friendly and fulfil the requirements of the Food and Drug Administration (FDA) and the European Medicines Agency (EMA) were used for the development of sustainable coatings. Their advantage is that they consist of natural compounds that can be taken orally and are therefore suitable for external use and safe for humans and the environment. By choosing erythritol, gelatin or collagen with propolis or alginate fibres and by varying the process parameters without using a catalyst or hydrophilic silicone emollients a synergistic approach with natural compounds was achieved. The aim of this work was, as mentioned

- to determine the possibility of modifying yarn with biopolymers
- to determine formulations that achieve a pleasant textile feel (hand value)
- to process the yarns using the dip-coating method
- determine the basis for daily processing for e.g. antibacterial, antifungal, etc. based on the processing and characterisation methods carried out
- reflect on the possible application of the samples based on the processing and characterisation methods carried out
- that the modifications carried out can be an ecological alternative to methods such as sol-gel

The optimal formulations were selected; the samples were processed by dip coating and as such can serve as a basis for further refinement of the formulation with the aim of wider application in medicine.

Commercially available polyester yarn served as the basis for the development of the formulations. Regenerated cellulose fibres will certainly be used in the future to contribute to even better sustainability, the protection of human health and the environment.

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