Biocomposite filaments reinforced with natural-origin fibres for specialised biomedical applications

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ABSTRACT – REZUMAT

Biocomposite filaments reinforced with natural-origin fibres for specialised biomedical applications

In this study, biocomposite filaments based on alginic acid (sodium salt) reinforced with cotton fibres (length of 2 ± 0.5 mm) were developed. The polymeric matrix was prepared using alginic acid at a concentration of 1%, while cotton fibres were incorporated at concentrations of 0.5%, 1%, and 1.5%. The synthesis of biocomposite filaments was carried out using the wet spinning technique employing a mechanically actuated syringe, followed by crosslinking using a 1% calcium chloride solution.

The obtained filaments were initially characterised from morphological and chemical perspectives using scanning electron microscopy (SEM), energy-dispersive spectroscopy (EDS) and Fourier Transform Infrared Spectroscopy (FT-IR). The SEM results demonstrated that the obtained alginate filament has a dense, homogeneous structure with certain folds resulting from the wet-spinning process. Additionally, the reinforcement with cotton fibres forms a 3D network within the matrix and significantly modifies the porosity of the alginate filaments. Regarding the chemical composition of the filaments, the EDS analysis revealed that the predominant elements are carbon and oxygen, thus indicating the organic nature of both the alginate matrix and the reinforcing agent. FTIR analysis enabled the identification of the main functional groups corresponding to characteristic absorption bands from the fingerprint region of each precursor material, for all composite materials. Afterwards, the absorption capacity of all filaments, with/without cotton fibres, was tested in physiological serum. The results revealed statistically significant differences as a function of the cotton fibres incorporation ratio, highlighting the impact of composition on the hydrophilic behaviour of the filaments. The development of composite filaments based on alginic acid is a crucial aspect in obtaining innovative hybrid materials characterised by low cost, biocompatibility, and biodegradability. These structures can be effectively implemented in various biomedical applications, including controlled and targeted drug delivery, soft tissue engineering, and the development of advanced dressings for wound treatment.

Keywords: *alginic acid, hybrid biomaterials, natural fibres reinforced, wound dressings.*

Filamente biocompozite ranforsate cu fibre de origine naturală pentru aplicații biomedicale avansate

În acest studiu au fost dezvoltate filamente biocompozite pe bază de alginat de sodiu, ranforsate cu fibre de bumbac cu lungimea de 2 ± 0,5 mm. Matricea polimerică a fost obținută dintr-o soluție de alginat de sodiu cu concentrația de 1%, iar fibrele de bumbac au fost adăugate în proporții de 0,5%, 1% și 1,5%. Filamentele au fost fabricate prin tehnica de filare umedă (wet spinning), utilizând o seringă acționată mecanic, după care au fost reticulate într-o soluție de clorură de calciu 1%.

Filamentele obținute au fost caracterizate inițial din punct de vedere morfologic și chimic prin microscopie electronică de baleiaj (SEM), spectroscopie de dispersie a energiei razelor X (EDS) și spectroscopie în infraroșu cu transformată Fourier (FT-IR). Imaginile SEM au evidențiat faptul că filamentul de alginat prezintă o structură compactă și relativ uniformă, cu anumite neregularități de suprafață generate în timpul procesului de filare. Totodată, introducerea fibrelor de bumbac a determinat formarea unei rețele tridimensionale în interiorul matricei polimerice și a influențat semnificativ gradul de porozitate al filamentelor.

Analiza EDS a arătat că principalele elemente prezente în compoziția filamentelor sunt carbonul și oxigenul, confirmând caracterul organic atât al matricei de alginat, cât și al fibrelor de bumbac utilizate ca material de ranforsare. Prin analiza FT-IR au fost identificate grupările funcționale caracteristice materialelor componente, evidențiindu-se prezența benzilor de absorbție specifice atât alginatului, cât și fibrelor de bumbac în structura biocompozitelor obținute.

În continuare, a fost evaluată capacitatea de absorbție a filamentelor în ser fiziologic. Rezultatele au evidențiat diferențe semnificative statistic între probe, în funcție de conținutul de fibre de bumbac, demonstrând că proporția materialului de ranforsare influențează comportamentul hidrofil și capacitatea de absorbție a filamentelor.

Dezvoltarea filamentelor compozite pe bază de acid alginic reprezintă un pas important în obținerea unor materiale hibride inovatoare, caracterizate prin costuri reduse, biocompatibilitate și biodegradabilitate. Datorită acestor caracteristici, ele pot fi utilizate în numeroase aplicații biomedicale, precum sistemele de eliberare controlată a medicamentelor, ingineria țesuturilor moi și realizarea de pansamente moderne pentru tratarea și regenerarea rănilor.

Cuvinte-cheie*: acid alginic, biomateriale hibride, materiale ranforsate cu fibre naturale, pansamente pentru vindecarea rănilor*

INTRODUCTION

The world of biomaterials, especially polymers, continues to have a significant impact on the field of biomedicine. The diversity of polymers and the different ways in which they can be used in scaffolds have developed considerably in recent years, offering active solutions to everyday problems. In the last decades, combinations of different scaffolds in a single solution have been investigated, showing great potential in wound healing and drug delivery systems, in particular in the fight against antibiotic-resistant pathogens.

The development of composite filaments based on biodegradable and biocompatible polymers has led to remarkable advances in regenerative therapies. These innovative materials are of great interest to both science and engineering, offering unmatched versatility and effectiveness across a wide range of biomedical applications (e.g., tissue engineering scaffolds for bone and cartilage regeneration, biodegradable sutures, controlled drug delivery systems, wound healing dressings, and nerve regeneration conduits) [1]. By harmoniously integrating with biological tissues and creating a favourable environment for cell proliferation and tissue regeneration, hydrogel-based therapies have demonstrated exceptional potential in addressing clinical challenges, particularly in wound healing [2]. Consequently, there is a growing demand for the development of next-generation dressings, patches, and bandages capable of effectively treating a wide spectrum of skin conditions, especially wounds and burns [3].

The reinforcement of pure alginate filaments with various fillers is widely recognised as an effective strategy for enhancing mechanical properties and liquid absorption capacity. A representative study in this area is that of Ahmad et al., who systematically investigated the incorporation of several types of fillers into alginate-based systems, including natural-based polymers (e.g., chitin, chitosan, cellulose, carrageenan, gelatin, and starch), synthetic polymers, carbon-based materials such as graphene oxide and carbon nanotubes, as well as inorganic fillers such as silver nanoparticles [4].

Recent literature reports the use of alginate as a polymeric matrix for the incorporation of various bioresorbable materials, functional composites, and smart hydrogels intended for biomedical applications; however, these approaches are often limited by drawbacks such as insufficient mechanical performance, tribological wear, or processing difficulties, thereby highlighting the innovative nature of the strategy investigated in the present study [5].

This study aims to develop sodium alginate filaments reinforced with natural cotton fibres, which could be successfully integrated into various biomedical applications, such as soft tissue engineering [6] or wound healing. These filaments are produced using the wet spinning technique, which involves extruding the polymer solution through a syringe and crosslinking it in a calcium chloride solution [7]. The quality of the process depends on several parameters, including the needle gauge, the length, density, and fineness of the cotton fibre, the actuation system of the syringe (manual or automated), and the ambient temperature [8]. Alginate, like starch [9], is a natural polysaccharide extracted from marine algae and is known for its gelling properties in the presence of calcium ions, forming three-dimensional hydrophilic networks (figure 1). The integration of natural fibres, such as cotton, into the alginate matrix can lead to enhanced mechanical properties of the biocomposite filaments [10]. Cotton, composed of 90–95% cellulose, has a fibrillar structure that promotes efficient interactions with the polymer matrix. Studies have shown that the addition of cotton fibres to alginate fibre structures can increase the Young's modulus up to 56.45 MPa, compared to 4.74 MPa for pure alginate fibres, highlighting a significant improvement in stiffness and mechanical strength [11].

Here, the integration of cotton fibres into the structure of alginate filaments is intended to increase the stiffness and tensile strength of the biocomposite materials, to provide superior structural stability, and to improve the overall material handling during final applications. The selection of this filler is based on the hypothesis that the specific properties of cotton (e.g., good mechanical strength, flexibility, biocompatibility, and excellent absorbency) can lead to the

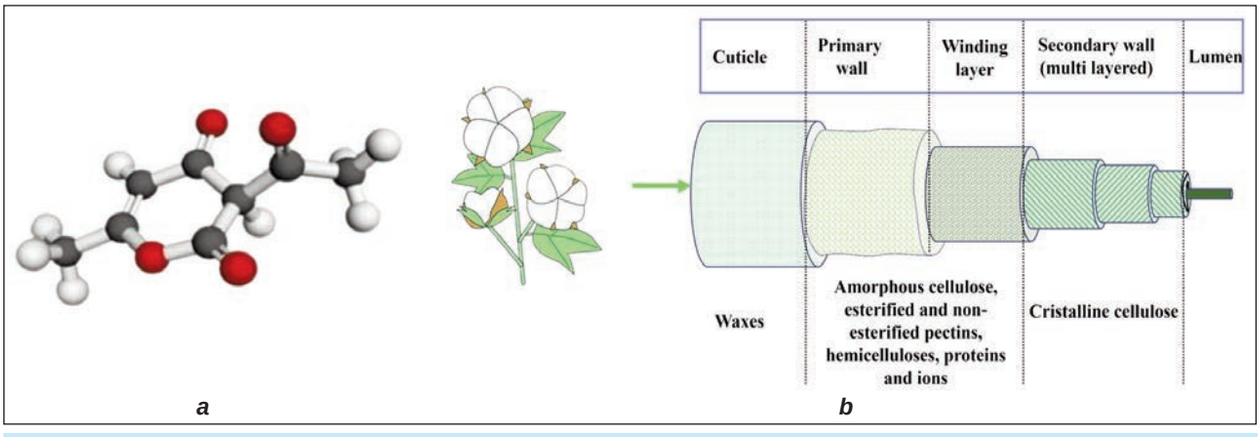

Fig. 1. Graphical representation of: *a* – alginate structure; *b* – structure of cotton fibre

development of biocompatible composite materials with superior performance compared to pure alginate [12].

MATERIALS AND METHODS

Synthesis of alginate-based biocomposite filaments reinforced with cotton fibres

In this experiment, two types of precursor materials were used: alginate powder (ALG) and cotton fibres (CTF), obtained by shredding cotton yarn. The cotton yarn spool was sourced from a textile company collaborating with the National Research & Development Institute for Textiles and Leather.

For the cotton fibres used in the biocomposite filaments, quality control measures were implemented to ensure batch-to-batch consistency. These included measuring fibre length and diameter to verify uniformity, inspecting for residual finishes or impurities that could affect filament formation, and monitoring moisture content to prevent degradation during storage. Such measures are essential for maintaining reproducible mechanical and structural properties of the resulting composite filaments.

Sodium alginate was supplied by Thermo Scientific, while the calcium chloride ($CaCl_2$) used for fibre crosslinking (density = 2.15 g/cm^3, M = 110.98 g/mol, granule size ≤ 7 mm, purity ≥ 93%) was purchased from Sigma-Aldrich.

Within the experiment, sodium alginate played the role of a precursor polymer, being water-soluble and allowing the formation of a viscous solution suitable for the wet-spinning process. Calcium chloride was used as a crosslinking agent in the coagulation bath, with Ca^{2+} ions interacting with the carboxyl groups of the alginate chains. Crosslinking occurs through an ion-exchange reaction, in which sodium ions are replaced by calcium ions, according to the "egg-box" mechanism, equation 1:

$$2NaC_6H_7O_6 + CaCl_2 \rightarrow Ca(C_6H_7O_6)_2 + 2NaCl \quad (1)$$

This reaction leads to the formation of a stable three-dimensional network, which instantaneously solidifies the extruded filament.

Table 1

SAMPLE CODIFICATION	
Sample code	**Sample description**
P0	CTF
P1	1%ALG
P2	1%ALG 0,5%CTF
P3	1%ALG 1%CTF
P4	1%ALG 1,5%CTF

To obtain both simple and composite filaments from sodium alginate, as well as filaments reinforced with cotton, the wet spinning technique was employed. A concentration of 1% ALG was used, while for the cotton-reinforced filaments, three different concentrations of cotton fibres were considered: 0.5% CTF, 1% CTF, and 1.5% CTF. Sodium alginate powder was dissolved in distilled water for 8 hours at a constant temperature of 30°C using a magnetic stirrer hotplate. The same procedure was followed for the preparation of biocomposite filaments reinforced with cotton fibres. The filaments were crosslinked in a 1% calcium chloride ($CaCl_2$) solution, in which they were immersed for 15 minutes. Afterwards, they were rinsed with distilled water and allowed to dehydrate at room temperature (figure 2).

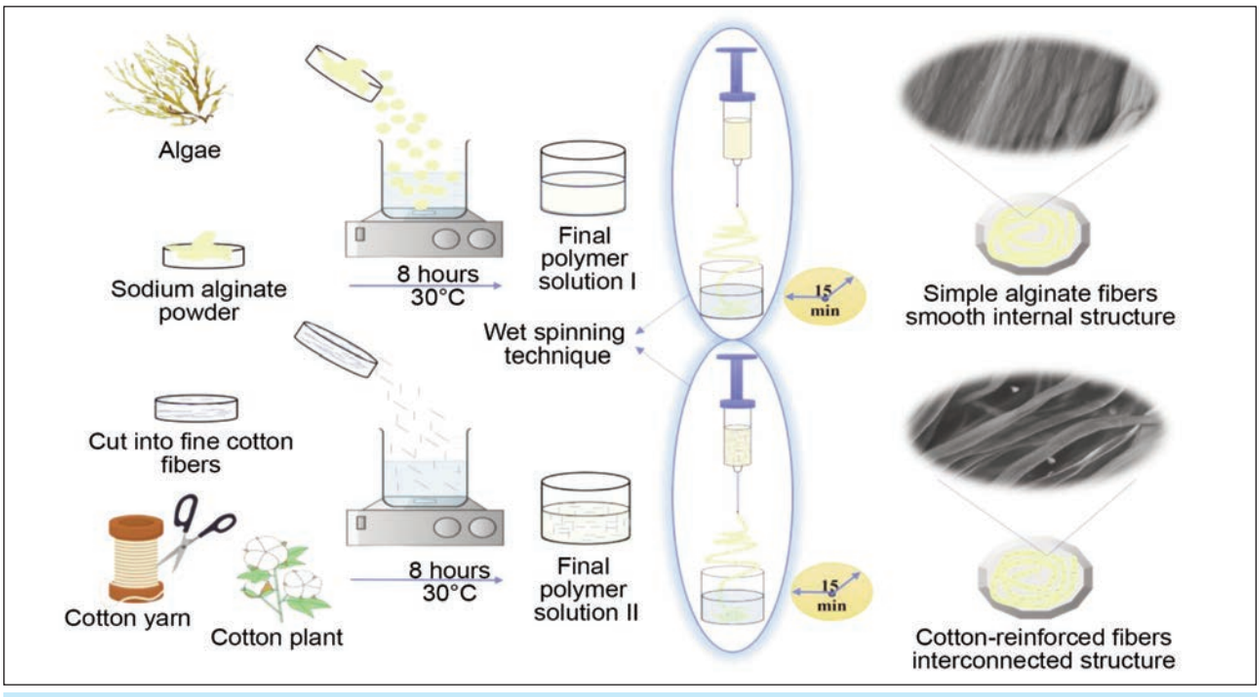

Fig. 2. Graphical representation of the process for obtaining simple alginate filaments and composite filaments reinforced with natural cotton fibres

The fibre spinning process was carried out by mechanically applying constant pressure to a syringe equipped with a 16G needle. For long-term preservation, it is recommended that the filaments be stored in distilled water, which should be replaced every 72 hours.

RESULTS AND DISCUSSIONS

Morphological characterisation by scanning electron microscopy (SEM)

The surface topography and morpho-compositional analysis of the alginate filaments and those reinforced with cotton fibres were performed by scanning electron microscopy (SEM) (Philips XL 30 ESEM TMP) coupled with energy-dispersive X-ray spectroscopy (EDS) (EDAX Sapphire spectrometer) [13].
SEM analysis for sample P1 showed that the sodium alginate biocomposite filaments revealed a smooth and uniform surface with no agglomerations or obvious pores. The fibre presents a linear and elongated structure, while the surface appears homogeneous and without significant roughness. This degree of smoothness is characteristic of pure alginate filaments. The absence of surface defects or fibril discontinuities suggests a stable spinning process with well-controlled parameters, leading to a continuous and structurally homogeneous filament [11]. Therefore, the observed morphology confirms the reliability and efficiency of the wet-spinning method employed.

With the addition of cotton fibres as a reinforcing agent, changes in fibre morphology and structure are observed. For sample P2, the surface is deeply textured, with folds and agglomerations. The cotton fibres are uniformly distributed inside and outside the long fibre, creating an interlocking system between the alginate matrix and the cotton fibres, which may contribute to increased resistance during final application. A non-uniformity in fibre distribution within the filament could lead to anisotropy of mechanical properties.
The surface of sample P3 is also rough, with visible fibres partially exposed, suggesting effective bonding and sufficient adhesion between the alginate matrix and the reinforcing agent.
The morphology of this fibre is in fact a combination of folds with a fibrous texture, which denotes that the internal structure is becoming more and more consolidated (figure 3).
Fibrillar continuity remains well preserved, however, and at this level of reinforcement, the P3 fibre shows no macroscopic discontinuities, i.e. no breaks or segregations of phases along the filament length are observed. The images suggest that most of the cotton fibres are firmly embedded in alginate, with the polymer matrix adhering around them, denoting a compatible interface (alginate is hydrophilic like cotton cellulose, facilitating hydrogen bond adhesion) [14].
In the case of sample P4, it is visible that the high content of cotton fibers that have penetrated the

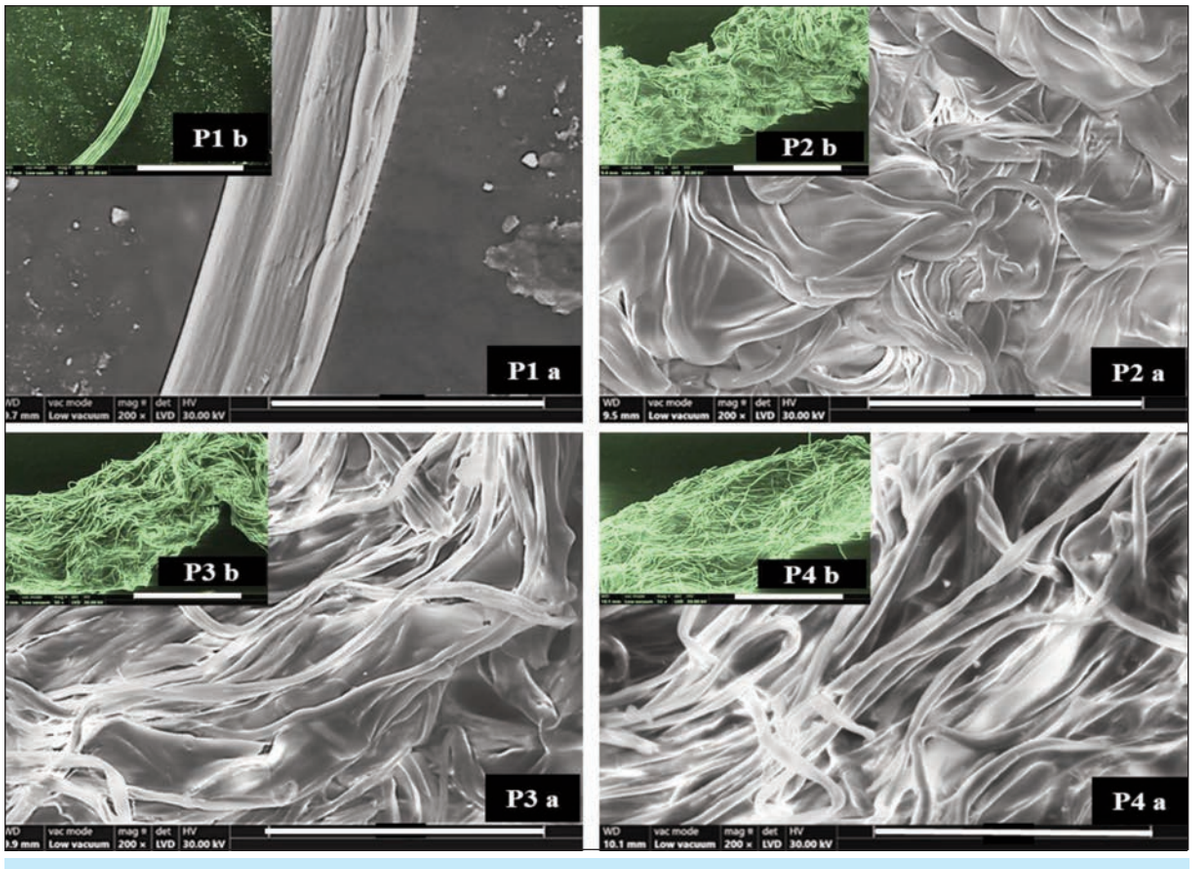

Fig. 3. Morphological evaluation of sodium alginate and composite fibres; scale bar: *a* – 300 µm; *b* – 1 mm

polymer matrix, forming a dense and pronounced network. The fibre surface is more uniform, unlike the other fibres, and has a less compact morphology. Despite the high amount of solid phase, the fibre retains its overall elongated shape, but with a higher porosity [15]. The CTF fibre dispersion in P4 is thus less uniform, although on a global scale cotton is still well dispersed; areas of higher cotton fibre density can be identified. These micro-agglomerations lead to the heterogeneous surface appearance and may indicate the existence of stress concentration points in the structure. In terms of fibrillar continuity, the P4 fibre remains overall continuous – no macroscopic fibre breaks or complete phase segregation are observed; the alginate forms a filament that binds the whole structure [16].

Determination of chemical composition by Energy Dispersive X-ray Spectroscopy (EDS)

The EDS spectrum shows that the predominant elements in P1 are carbon (C) and oxygen (O), derived from the organic matrix of alginate. This result is expected, since sodium alginate is a polymer rich in C and O (figure 4).

On the other hand, sodium (Na) appears in very small amounts, indicating that most of the initial Na^+ from sodium alginate was removed or replaced by Ca^{2+} during fibre formation. The literature reports that pure sodium alginate has an atomic ratio of C:O:Na ≈ 6:6:1, according to its chemical formula. In the case of the obtained calcium alginate, Na decreases substantially, while Ca becomes the characteristic crosslinking element in the polymeric network [17–20]. Additionally, traces of chlorine (Cl) can be detected in P1, which may be residual, resulting from insufficient washing.

Carbon and oxygen are the dominant elements in the composition of all samples, being the primary constituents of polysaccharides (both alginate and the cellulose from cotton). In sample P1 (only 1% alginate hydrogel), EDS analysis indicates a predominance of C and O due to the organic backbone of alginate. With the addition of cellulose fibres (P2–P4), the carbon and oxygen signals remain high or even increase slightly in relative proportion.

Thus, as the fibre content increases from P1 to P4, carbon and oxygen remain the most abundant elements, highlighting the organic nature of all the composites. The qualitative trend shows either maintenance or a slight increase in their relative percentages, considering that the addition of cotton is a rich source of C and O.

Structural investigations by FT-IR analysis

The bonding architecture, structure and environment of functional groups present for both the precursors, namely sodium alginate and cotton fibres, as well as the alginate filaments reinforced with cotton fibres, were analysed by Fourier Transform Infra-Red (FTIR) spectroscopy in attenuated total reflectance (ATR)

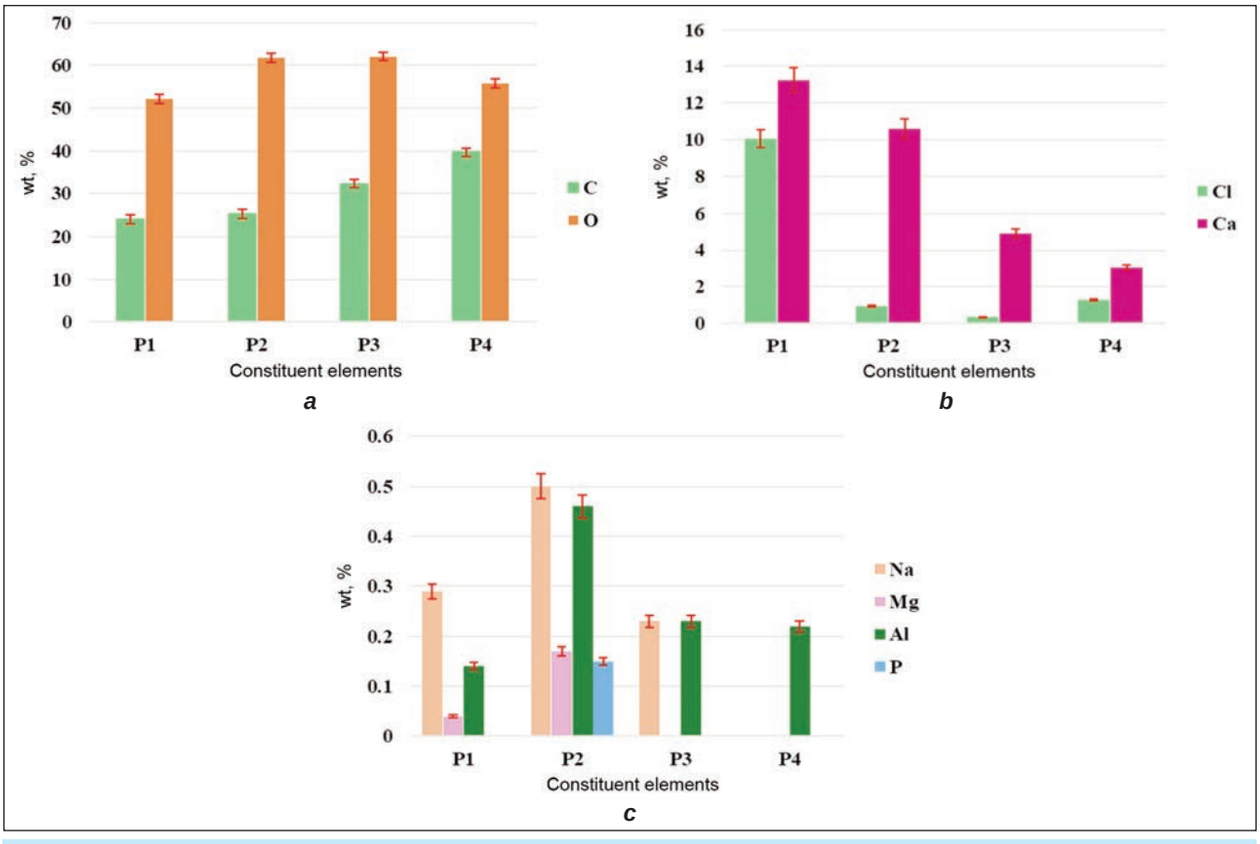

Fig. 4. The distribution of constituent elements for simple and composite ALG filaments: *a* – distribution of the main elements C and O; *b* – distribution of the elements Ca and Cl; *c* – distribution of the trace elements Na, Mg, Al and P

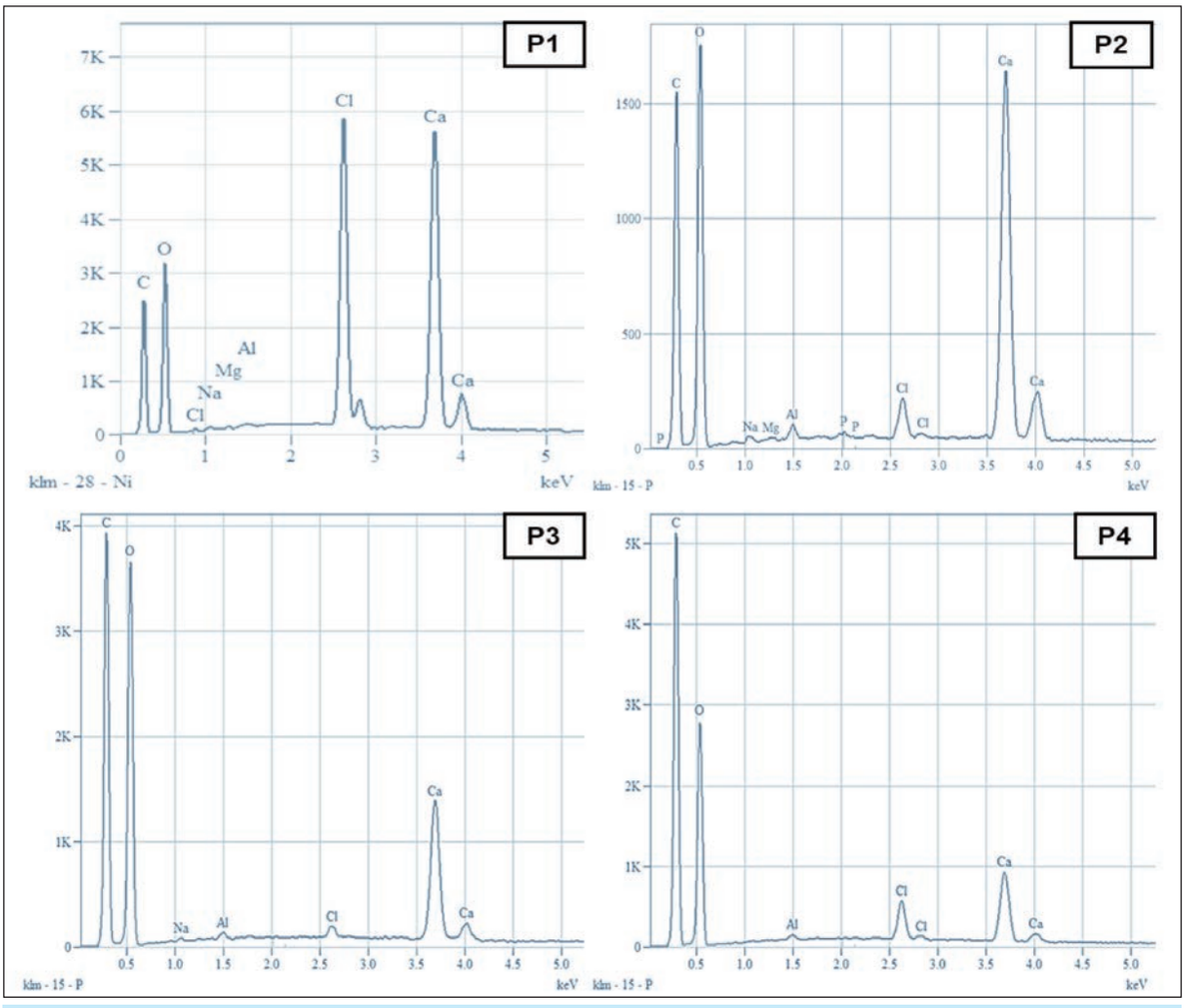

Fig. 5. The chemical composition of biocomposite filaments reinforced with natural cotton fibres

mode. The spectra were collected in the 4000 – 400 cm^{-1} wavenumber range. The IR fingerprints of all samples are presented in figure 6.

In the case of cotton fibres, a characteristic broad peak can be observed in the wavelength range of 3250–3500 cm^{-1}, corresponding to the –OH functional groups. Additionally, the absorption bands at 2900 cm^{-1} and 1630 cm^{-1} are associated with C–H stretching and –CO stretching vibrations, respectively. The peaks located at 1020 cm^{-1} and 1417 cm^{-1} were attributed to C–O stretching and C=C stretching vibrations, respectively. All these features confirm the highly cellulosic structure of cotton fibres.

For the pure alginate filament, a characteristic peak at approximately 1600 cm^{-1} was identified, which is attributed to the asymmetric stretching vibration of the COO^- group originating from the carboxylic acid salt. Another prominent peak appears around 3200 cm^{-1} and is assigned to the –OH group, arising from intermolecular hydrogen bonding. Two additional relevant peaks were observed at around 1030 cm^{-1} and 1100 cm^{-1}, corresponding to –CH stretching and –OH bending vibrations, respectively [11].

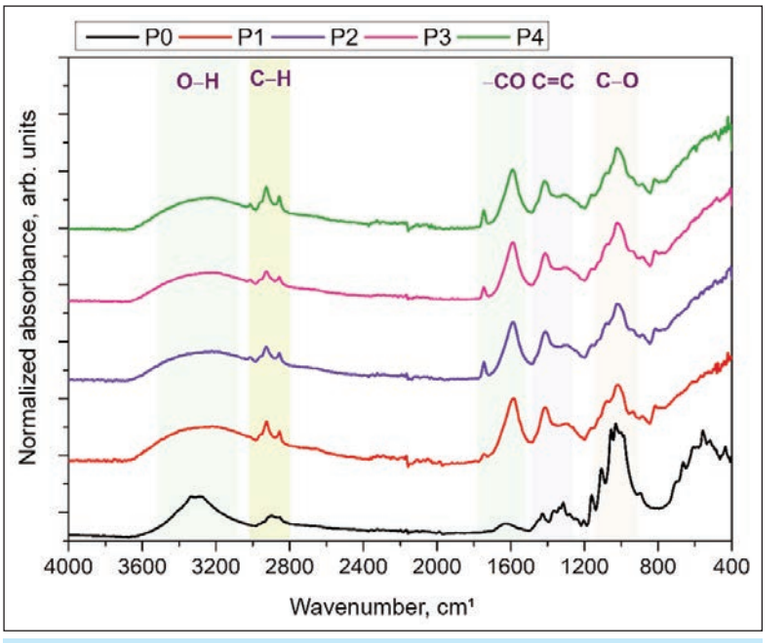

Fig. 6. Comparative FT-IR spectra of biocomposite filaments reinforced with natural cotton fibres

In the case of alginate filaments reinforced with cotton fibres, the FT-IR spectra confirmed a decrease in the intensity of the characteristic peaks near 3300 cm^{-1} and 2900 cm^{-1}, which can be attributed to the presence of cotton fibres within the alginate matrix. The formation of hydrogen bonds between the sodium alginate hydrogel and the cotton fibres, facilitated by hydroxyl and carbonyl functional groups, leads to a reduction in peak intensity as well as a shift in the corresponding spectral bands, as reported in other studies [12].

Absorption in liquids

Absorption was carried out in physiological serum (0.9% NaCl). The absorbance of physiological serum at the filament interface was investigated using the ASTM D570 standard. The sample was first dried in an oven for 4 h at 70°C, then weighed (M_i) on an analytical balance with a minimum division of 0.1 mg. Preconditioned samples were then placed in physiological saline for 24 hours at room temperature and pressure. After 24 hours, the samples were removed, blotted with filter paper to remove excess serum and then reweighed (M_f). After determination of the dry and wet mass of the fibres, the water absorption capacity was calculated using equation 2 [21]. The statistical analysis for the absorption capacity was performed using the one-way ANOVA test (post hoc Tukey's multiple comparisons test); the p values below 0.05 are considered to be statistically significant:

$$Absorption\ of\ physiologic\ serum\ (\frac{g}{g}) = \frac{M_f - M_i}{M_i} \times 100 \quad (2)$$

High absorption capacity is the main characteristic of alginate filaments. One of the key aspects of scaffolds is their water content and their ability to retain water. Wound exudates must be absorbed in order to promote the healing process by controlling cellular dehydration and stimulating both angiogenesis and collagen synthesis. Proper moisture control accelerates the healing rate, reduces pain, and provides protection against wound infections. Wound exudates can separate tissue layers, leading to delayed healing. Therefore, absorption is a crucial property of alginate filaments used as scaffolds for various biomedical applications. Table 2 presents the values obtained from weighing the samples using the analytical balance, both before absorption (M_i) and after absorption (M_f).

Serum absorption decreases with increasing cotton concentration: the 1.5% CTF samples retrieved the lowest serum absorption, while the 1% CTF one the highest. On the other hand, the serum uptake of cotton-reinforced alginate filaments is statistically lower than that of pure alginate filaments. Overall, the decrease in absorbency was owed to the use of CTF reinforcement, a material known for its hydrophilic character and ability to absorb fluids [11].

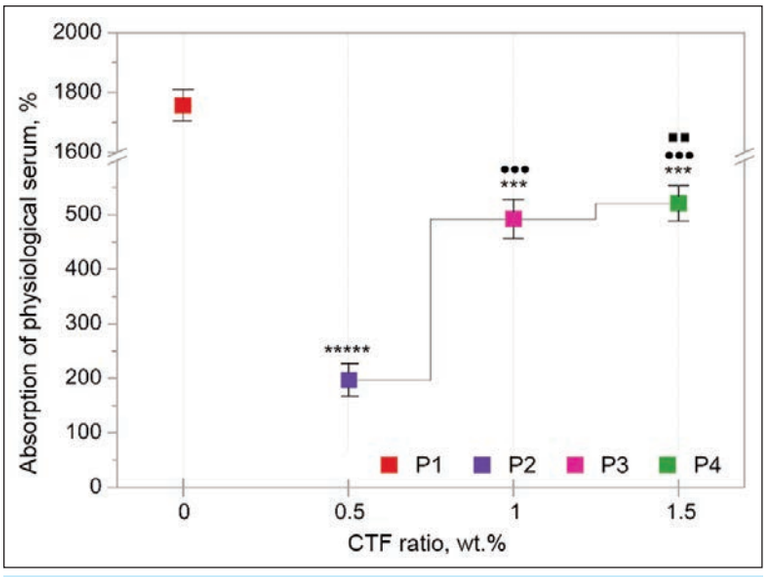

Fig. 7. Absorption of physiological serum of simple and biocomposite filaments after 24 hours of immersion. The ALG material was used as reference (P1) sample; data analysis was based on mean ± SD (n = 5; ***** $p < 0.0001$, *** $p < 0.001$ vs. P1; ●●● $p < 0.001$ vs. 1% ALG 0.5%CTF; ■■ $p < 0.05$ vs. 1%ALG 1%CTF)

CONCLUSIONS

The feasibility of developing alginate/cotton biocomposite materials and filaments through the proposed preparation mechanism was confirmed.

All composite filaments maintained a proper morphology, exposing good fibrillar continuity and demonstrating the viability of the wet-spinning process even in the presence of the disperse phase.

The alginate-cotton interface exhibited satisfactory adhesion, supported by the

Table 2

ABSORPTION OF PHYSIOLOGIC SERUM AFTER 24 H OF IMMERSION				
Sample code	**Sample description**	**M_i (g)**	**M_f (g)**	**Absorption of physiologic serum (%)**
P1	1% ALG	0.005	0.094	1780
P2	1% ALG 0,5%CTF	0.0078	0.0231	196
P3	1%ALG 1%CTF	0.0081	0.0485	499
P4	1%ALG 1,5%CTF	0.0123	0.075	510

common hydrophilic nature of the two components, favourable for hydrogen bond formation. The EDS analysis revealed the consistent presence of calcium, carbon and oxygen in the matrix.

Further, the FT-IR analysis confirmed the successful incorporation of cotton fibres into the alginate matrix, as evidenced by the presence and modification of characteristic spectral bands. The observed changes in peak intensity and slight band shifts indicated the formation of intermolecular hydrogen bonds between alginate and cotton fibres, demonstrating good interfacial compatibility and structural stability of the composite filament.

Overall, the alginate filaments exhibited statistically high serum absorption, a key requirement for biomedical scaffolds. The addition of cotton fibres reduced the absorption capacity compared to pure alginate, with higher cotton content leading to lower uptake; however, the reinforced filaments still retain adequate absorbency suitable for wound-healing applications.

Thus, alginate/cotton biocomposite filaments are promising materials for areas such as wound care, medical textiles or applications with flame retardant requirements. However, for targeted applications in the biomedical field, future research directions are envisioned in terms of mechanical behaviour investigation, biocompatibility assessments, along with antimicrobial evaluations, which will lead to an optimised modulated composition of the composite materials.

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