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Sol-gel/Ag coating and oxygen plasma treatment effect on synthetic wound fluid sorption by non-woven cellulose material

Učinek prevleke sol-gel/Ag in obdelave s kisikovo plazmo na absorpcijo sintetičnih izločkov iz ran pri netkanem celuloznem materialu

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Abstract

Non-woven cellulose material was functionalized using two techniques, i.e. the coating with AgCl via sol-gel and oxygen plasma. The treatment effects were studied regarding the wound fluid adsorption potential using physiological saline, synthetic exudate and synthetic blood. Plasma treatment was most efficient since a significant improvement by absorbency rate and capacity was evident, less pronounced in case of synthetic blood. The combination of both treatments showed a similar trend, while the effects were less prominent, but still sufficient by managing fluid-associated as well as infected wounds.
Keywords: non-woven cellulose fabric, sol-gel, oxygen plasma, absorption, synthetic wound fluids

Izvleček

Netkani celulozni material je bil funkcionaliziran z uporabo dveh tehnik, tj. postopkom sol-gel in s kisikovo plazmo. Obdelave so bile okarakterizirane glede na potencial adsorpcije izločka iz rane z uporabo fiziološke raztopine, sintetičnega eksudata in sintetične krvi. Plazemska obdelava je bila učinkovitejša, saj je bistveno izboljšala vpojnost in zadrževanje testiranih tekočin. Med testiranimi tekočinami je izboljšanje hidrofilnosti najmanj izrazito pri sintetični krvi. Kombinacija obeh obdelav je pokazala podoben trend absorpcije, le da je ta bila manjša, a še vedno zadostna za oskrbo ran z veliko izločka in tudi za okužene rane.
Ključne besede: celulozna vlaknovina, sol-gel, kisikova plazma, absorpcija, sintetični izločki iz ran

1 Introduction

A wound dressing, designed for optimal healing, should provide antimicrobial protection along with wound drainage and toxic components removal [1, 2]. Silver is known as an excellent antimicrobial agent [2–5], applied in many wound dressing products [2]. Plasma treatment is one of the most versatile techniques in polymer surface modifications [6] used for increasing wettability [7] and adhesion [8], while the best results are observed when using oxygen gas [9]. In our previous studies [10], we combined safe silver binding on cellulose substrate and oxygen plasma modification aiming at achieving simultaneously antimicrobial properties along with improved hydrophilicity. The silver-containing samples showed minimal silver release while providing safe antimicrobial activity towards four most common present bacteria in wounds. Hydrophilicity was significantly improved when plasma was applied, as

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determined using water as the most common used absorbate. In this study, the absorption properties were determined using physiological saline solution, synthetic exudate and synthetic blood, simulating a wound fluid model.

2 Materials and methods

2.1 Materials

A non-woven fabric composed of regenerated cellulose fibers with surface mass 175 g/m^2 and the thickness 1.7 mm (purchased from KEMEX, The Netherlands) was sol-gel coated and oxygen plasma treated.

2.2 Preparation procedure

The samples studied are denoted as N- for non-treated cellulose non-woven, S – for sol-gel silver coated, P – for oxygen plasma modified and SP – for sol-gel silver coated and after that oxygen plasma treated.

2.2.1 Sol-gel silver binding

Silver nano-particles in silver chloride form, iSys Ag (CHT, Germany) in combination with iSys LTX (CHT, Germany) as organic-inorganic binders, were used to achieve an antimicrobial effect of the used materials. Kollasol CDO (CHT, Germany) was used as a wetting agent. Kollasol is a hydrophilic silicone surface-active substance mixed with higher alcohols.

Firstly, the water solution of iSys LTX (5 g/L), iSys Ag (5 g/L) and Kollasol (0.7 g/L) was prepared. Viscose non-woven samples were impregnated in solution with bath ratio 1:30 at room temperature for 1 h. After treatment, viscose was wrung-out with a foulard (Werner Mathis Ag, Switzerland) with a pressure of 4 bar between cylinders and their speed of rotation at 0.5 m/min, oven dried in a stretched state at 80 °C, and additionally condensed for 1 min at 150 °C (Werner Mathis Ag, Switzerland).

2.2.2 Oxygen plasma treatment

Before plasma treatment, the samples were air-conditioned at 20 °C and 65% RH for 24 h. The samples were treated with oxygen plasma in a discharge chamber. The discharge chamber was a spherical cylinder with an inner diameter of 36 cm and the height of 30 cm. Plasma was created with an inductively coupled RF (radio frequency) generator, operating at a frequency of 27.12 MHz and output power of about 500 W. During the experiment, the pressure was fixed at 75 Pa. A more detailed description of the treatment procedure is written elsewhere [11,12]. The samples were exposed to oxygen gas for 10 min.

2.3 Capillary rise method

To simulate real wound fluid-handling, three test liquids were used, i.e. physiological saline solution, synthetic exudate and blood. The composition and physical properties of used liquids are described in our previous study [13].

The capillary rise method was used as a modified experimental procedure based on Washburn equation [14]. It enables the characterization of porous solid and also fabric with regard to their wettability. The samples in circular shape (2.5 cm in diameter; 0.087 g) were placed in the Krüss K12 Tensiometer (Krüss GmbH, Germany). All measurements were performed at constant temperature of 20 °C. For a more detailed description of the experimental technique, the readers are referred to [13].

As an experimental result, the weight increase (m) as a function of time (t) is observed. The amount of liquid uptake in equilibrium represents the absorbency capacity. By determining the slope of the linear part of these plots, the quantity m^2/t is obtained indicating the absorbency rate. At least ten single measurements were performed for each sample with each test liquid.

3 Results and discussion

Fig. 1 presents the absorbency rate of physiological saline, synthetic exudate, and blood solution in non-treated (N), oxygen plasma (P), sol – gel/Ag (S), and sol – gel/Ag and plasma (SP) treated samples. The curves in Fig. 1 are presented only for 100 s of monitoring the wetting process.

The slowest absorbency rate was evident by non-treated sample (N) irrespective of used wound relevant fluids. The complete wetting for physiological saline and synthetic exudate solutions occurred only after 300 s and the amount of liquids' uptake in equilibrium amounted 1.2 g and 1.3 g for saline and exudate solutions, respectively. The blood rise curve reached the plateau only just after 1200 s and the equilibrium uptake amounted 1 g.

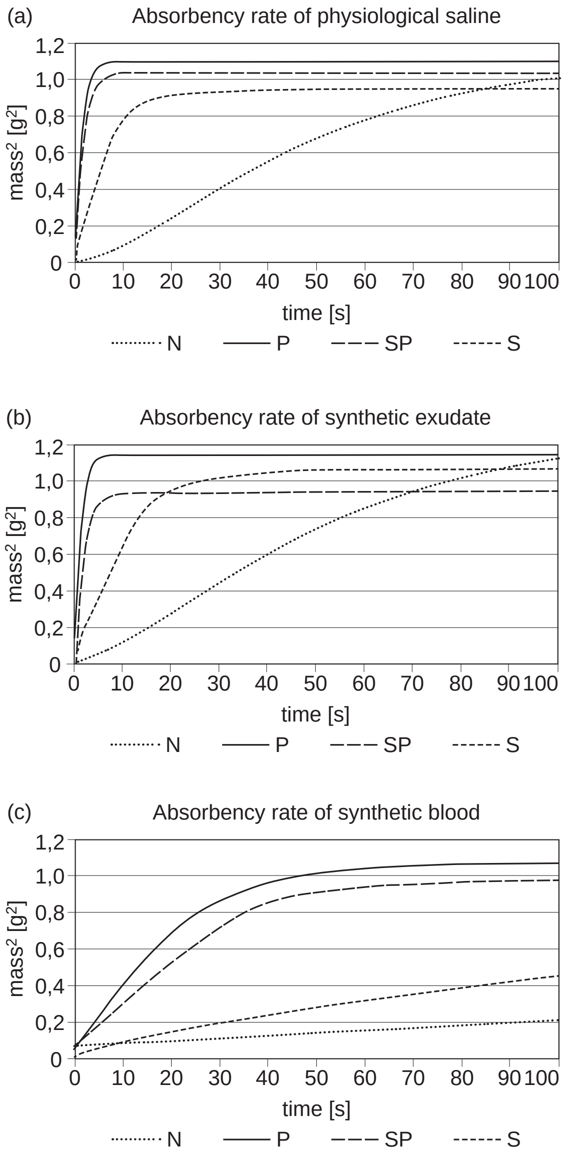

Figure 1: The square mass data of physiological saline (a), synthetic exudate (b), and blood solution (c) rise on non-treated (N), oxygen plasma (P), sol – gel /Ag (S), and sol – gel/Ag and plasma (SP) treated samples

Plasma treatment significantly improved the hydrophilicity since the equilibrium was obtained in a much shorter time, i.e. 10 s for saline and exudate and 66 s for blood. The plasma treated sample (P) was able to uptake above 1 g of all tested wound relevant fluids.

Among treated materials, the sol-gel/Ag sample showed the lowest wetting rise curves by all tested liquids. By saline solution the complete wetting occurred within 20 s, by exudate the plateau was obtained within 40 s, while for blood the equilibrium was established no sooner than within 300 s. The sol-gel/Ag sample (S) was able to absorb about 1 g of tested liquids in equilibrium.

The absorbency rate for antimicrobial and plasma modified sample (SP) was slightly lower compared to the plasma treated sample (P). The complete wetting for all used liquids occurred within the same time as by the plasma treated samples, while the equilibrium uptake amount was up to 1 g.

The results presented in Fig. 1 demonstrated the effects of the used treatments on the absorbency rate and capacity using wound relevant fluids. Plasma treatment evidenced the biggest effect due to incorporation of polar functional groups and/or cleaning and/or etching. The sol-gel/Ag treated sample showed the lowest absorbency rate and capacity due to organic-inorganic origin of sol-gel components influencing the inter-molecular interactions between liquid probes and solid surfaces. Absorbency properties after applied plasma treatment onto the sol-gel/Ag samples (SP) was also significantly noticeable, but the effect on the rate and capacity was less evident compared to the plasma treated sample (P). A faster absorbency rate by physiological saline and synthetic exudate solutions compared to synthetic blood for all tested samples raised due to different liquids' physical (e.g. viscosity and surface tension) and chemical (e.g. composition, size and shape of molecules) properties.

4 Conclusions

A non-woven cellulose material was modified using two processes, i.e. sol-gel/Ag and oxygen plasma treatment to simultaneously gain both desired properties for a dressing appropriate for healing infected and exudating wounds. Sorption properties were evaluated using synthetic solutions simulating wound fluids.

Plasma treatment, when applied as single procedure as well as in two-step procedure significantly improved the absorbency rate and capacity for all tested liquids. Although the plasma effect was less evident when using synthetic blood, the obtained results indicate the importance of testing in a simulated real environment before being suitable for application.

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References

1. PETRULYTE, Salvinija. Advanced textile materials and biopolymers in wound management. *Danish Medical Bulletin*, 2008, **55**(1), 72−77.
2. LEAPER, David J. Silver dressings: their role in wound management. *International Wound Journal*, 2006, **3**(4), 282−294, doi: 10.1111/j.1742 481X.2006.00265.x.
3. RAJAR, Barbara, SUKIČ, Neža, KREBELJ, Sandra, MALNIG, Andrea, ČUBRILOVIĆ, Milenko, TOMŠIČ, Brigita, GORJANC, Marija, SIMONČIČ, Barbara. Preparation of multifunctional repellent and antimicrobial active polyamide 6 fabric pretreated with oxygen plasma. *Tekstilec*, 2016, **59**(1), 15–27, doi: 10.14502/Tekstilec2016.59.15-27.
4. LOGAR, Nina, KLEMENČIČ, Danijela, TOMŠIČ, Brigita, PAVKO ČUDEN, Alenka, SIMONČIČ, Barbara. Tailoring of a dual-active antibacterial coating for polylactic acid fibres. *Tekstilec*, 2016, **59**(4), 289–297, doi: 10.14502/Tekstilec2016.59.289-297.
5 LIU, Xin, LIN, Tong, GAO, Yuan, XU, Zhiguang, HUANG, Chen, YAO, Gang, JIANG, Linlin, TANG, Yanwei, WANG, Xungai. Antimicrobial electrospun nanofibers of cellulose acetate and polyester urethane composite for wound dressing. *Journal of Biomedical Materials Research Part B: Applied Biomaterials*, 2012, **100**(6): 1556−1565, doi: 10.1002/jbm.b.32724.
6. *Plasma Technologies for Textiles*. Edited by R. Shishoo. Boca Raton : CRC Press, Cambridge : Woodhead Publishing. The Textile Institute, 2007, 322.
7. HOLLÄNDER, Andreas, THOME, Jörg, KEUSGEN, Michael, DEGENER, Inke, KLEIN, Wolfgang. Polymer surface chemistry for biologically active materials. *Applied Surface Science*, 2004, **235**(1), 145−150, doi: 10.1016/j.apsusc.2004.05.132.
8. GLOCKER, A. David, SHAH, S. Ismat. *Handbook of thin film process technology*. Bristol : Institute of Physics Publishing, 1995.
9. CARLSSON, CM Gilbert, STROEM, Goeran. Reduction and oxidation of cellulose surfaces by means of cold plasma. *Langmuir*, 1991, **7**(1), 2492−2497, doi: 10.1021/la00059a016.
10. PIVEC, Tanja, PERŠIN, Zdenka, KOLAR, Mitja, MAVER, Tina, DOBAJ-ŠTIGLIC, Andreja, VESEL, Alenka, MAVER, Uroš, STANA-KLEINSCHEK, Karin. Modification of cellulose non-woven substrates for preparation of modern wound dressings. *Textile Research Journal*, 2014, **84**(1), 96−112, doi: https://doi.org/10.1177/0040517513483855.
11. VESEL, Alenka, SEMENIČ, Tomaž. Etching rates of different polymers in oxygen plasma. *Materials and Technology*, 2012, **46**(3), 227-231.
12. VESEL, Alenka. XPS study of surface modification of different polymer materials by oxygen plasma treatment. *Informacije Midem–Journal of Microelectronics Electronic Components and Materials*, 2008, **38**(4), 257-265.
13. PERŠIN, Zdenka, DEVETAK, Miha, DREVENŠEK OLENIK, Irena, VESEL, Alenka, MOZETIČ, Miran, STANA-KLEINSCHEK, Karin. The study of plasma's modification effects in viscose used as an absorbent for wound-relevant fluids. *Carbohydrate Polymers*, 2013, **97**(1), 143−151, doi: 10.1016/j.carbpol.2013.04.045.
14. WASHBURN, W. Edvard. The dynamics of capillary flow. *Physical Review*, 1921, **17**, 273−283.