

SOLUTION BLOWN OF PLA NANOFIBER CONTAINING OZONATED MORMODICA OIL AND ITS MICROCAPSULES TO OBTAIN ANTIBACTERIAL MEDICAL TEXTILES SURFACES

PEKTAŞ KORAY^{1*}, BALCI ONUR¹ AND ORHAN MEHMET²

¹ Kahramanmaraş Sütçü İmam University, Faculty of Engineering and Architecture, Textile Engineering Department, Avşar Campus, 46100, Onikişubat/ Kahramanmaraş, Turkey

² Uludağ University, Faculty of Engineering and Architecture, Textile Engineering Department, Görükle Campus, 16059, Nilüfer/ Bursa, Turkey

ABSTRACT

In the scope of the study, it was aimed to obtain antibacterial nanofiber surfaces containing Momordica oil, its ozonated oil form and its microcapsules forms. First of all, Momordica oil was exposed to ozone gas for 135 min. After that, crude and ozonated mormodica oil were microencapsulated by using simple coacervation. Subsequently, %10 PLA polymer solution were prepared and used for obtaining PLA nanofiber surface by using solution blowing spinning. Besides, PLA polymer solution were mixed with crude mormodica oil, ozonated mormodica oil and their microcapsules forms and then these solutions were spun by using solution blowing spinning. Obtained ozonated oil, microcapsules and nanofiber surfaces were characterized via measurement of total unsaturated fatty acid amount in the oils, scanning electron microscope, FT-IR analysis and antibacterial activity test. The data showed that mormodica oil were ozonated. Microencapsulation process was done successfully and obtained nanofiber containing mormodica oil and its microcapsules. Moreover antibacterial activity showed that mormodica oil and ozonated mormodica oil showed antibacterial activity against to *S.aureus* and *E.coli* bacteria according to the disc diffusion method. The nanofiber surfaces containing ozonated oil and its microcapsules showed antibacterial activity against to *S.aureus* and *E.coli* bacteria according to the ASTM E 2149-01 method. As a result, it was obtained biodegradable nanofiber containing microcapsules and showing antibacterial activity.

KEYWORDS

Solution blowing spinning; PLA; Ozonated oil; Microencapsulation; Antibacterial activity.

* Corresponding author: Pektaş K., e-mail: koraypektas@ksu.edu.tr

INTRODUCTION

Recently, the ozone (O₃) is applied to treat many illnesses such as cellulite, burnt, ulcer, chronic wounds, immune system illnesses etc. On examining these treatments, it is seen that the ozone is used both directly and ozonated vegetable oils in treatment of illnesses. However, comparing to ozone gas and the ozonated water, the ozonated oils have an advantage that the ozone is bonded to oil via unsaturated fatty acids. Thus, the ozone could be stored as ozonide and its effect goes a long [1-3]. The ozonides, which show anti-bacterial and anti-fungal activity, carry O₂ into lesion without sparking off skin irritation [4].

On the other hand, the microencapsulation is a preferable method to transfer ozonated oil onto textile surfaces. The microencapsulation is a caging method that liquid or solid particles, which are

located small droplets, are hindered in a thin film. The microencapsulation is formed by many methods such as in-situ polymerization, interfacial polymerization, coacervation, spray drying etc. [1,5]. But the coacervation methods are common to encapsulate oils among them [6].

Solution blown technique is a spinning method that inspired from both meltblown and electro spinning method, generate micro and nanofiber surfaces [7]. On examining literature on solution blowing spinning, there are much more studies on nanofiber spinning while there are a few studies on medical textile surfaces [8-11].

In this study, it was aimed to obtain biodegradable antibacterial nanofiber surfaces containing Momordica oil, its ozonated oil form and its microcapsules forms. To get these functionality, mormodica oil, ozonated mormodica oil and their microcapsules were mixed PLA polymer solution

and spun together. After that, a set of tests and analyses were employed both mormodica oil and obtained nanofiber surface.

EXPERIMENTAL

Materials

In the scope of this study, mormodica oil and its ozonated form were used as an active material. Arabic gum was used as shell material for microencapsulation process. PLA polymer was used for obtaining biodegradable nanofiber surfaces. Chloroform was used for dissolving the PLA polymer. 30 g/m² Polypropylene non-woven surfaces was used as a ground surface to collect PLA nanofiber.

Methods

Crude mormodica oil were exposed to ozone gas for 135 min in a glass reaction column and then ozonated mormodica oil obtained (Figure 1).

As for the microencapsulation of the crude and ozonated mormodica oil, simple coacervation method was used and flow chart of the microencapsulation process was given in Figure 2.

Solution blowing spinning was used for obtaining PLA nanofiber. With this purpose, 10g PLA polymer was dissolved in the chloroform (100ml) for 2 hours at 60 °C. Then the polymer solution was cooled to room temperature. After that solution blowing spinning parameters were adjusted and PLA nanofiber were obtained. To obtain PLA nanofiber

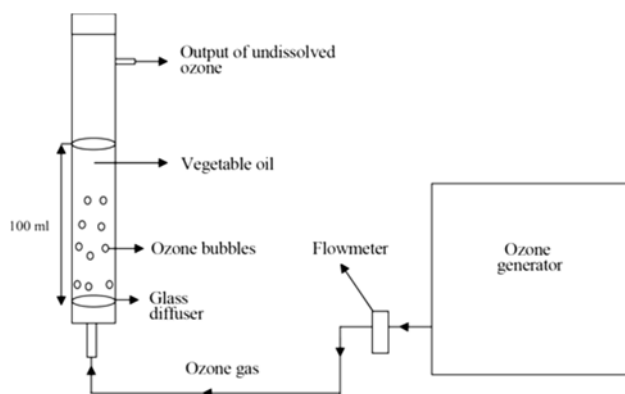


Figure 1. Schematic drawing of the ozonation process.

containing oil and microcapsules, 1 ml oil or microcapsule solution poured into the 20 ml PLA polymer solution. Then fiber spinning containing oil or microcapsules were employed. Fiber spinning parameter of the solution blowing spinning were given below.

- Solution feeding rate: 10ml/h.
- Air pressure: 2 bar
- Working distance: 37 cm
- Working time: 30 min.
- Distance between inner and outer nozzle: 2 mm

As for the characterization of the oils, total unsaturated fatty acid amount was determined by using GC and FT-IR analyses was done to observe change of the spectrum after ozonation process. Besides, disc diffusion method was used for investigating antibacterial activity of the oils. SEM images were taken for observe nanofiber morphology. Antibacterial activity of the nanofiber surfaces was analyzed according to the ASTM E 2149-01.

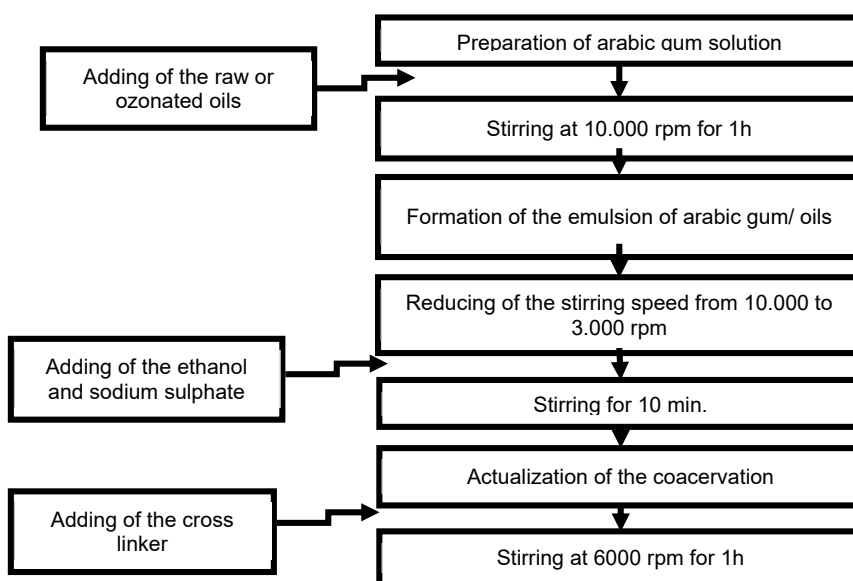


Figure 2. Flow chart of the microencapsulation process.

RESULTS AND DISCUSSION

Total unsaturated fatty acid amounts of the oil were given in Table 1.

Total unsaturated fatty acid amount of the oils showed that unsaturated fatty acid amount decreased after ozonation process. Because the bound of the =C-H in the crude oil were broken and replace C-O bound. during the ozonation process. To support this hypothesis, FT-IR spectrum of the oil were investigated. FT-IR spectrum of the oil (Figure 3) showed that C-O bound was seen at 1100cm^{-1} after ozonation process and it was proof of the ozonation of the mormodica oil was done successful.

On examining of the antibacterial activity of the crude and ozonated mormodica oil (Table 2), it was seen that both of them had antibacterial activity against to both gram negative (*E.coli*) and gram positive (*S.aureus*) bacteria with different inhibition zone. Besides, it was seen that antibacterial activity of the ozonated mormodica oil was higher than the crude one.

After characterization of the mormodica oil and ozonated one, PLA nanofiber surface were observed via SEM images. Upon observing of the PLA nanofiber morphology, it was seen that ground non-woven surfaces had micrometer fiber diameter while crude PLA fiber, containing oil and microcapsules ones had nanometer fiber diameter but some beads formations were observed. It was thought that nanofiber spinning parameter could change to hinder bead formations. Moreover, it was seen that microcapsules in the PLA nanofiber were seen as spherical shape.

Table 1. Total unsaturated fatty acid amount of the oils.

Unsaturated fatty acid (%)	Mormodica oil	Ozonated mormodica oil
Oleic acid	42,457	11,413
Linoleic acid	38,949	3.309
Total unsaturated fatty acid amount	81,406	14,722

Table 2. Antibacterial activity of the crude and ozonated mormodica oil.

Oil Sample	Inhibition zone diameter (mm)	
	<i>S.aureus</i>	<i>E.coli</i>
Crude mormodica oil	100	100
Ozanated mormodica oil	160	130

Antibacterial activity of the polypropylene non-woven and nanofiber surface against to gram negative (*E.coli*) and gram positive (*S.aureus*) bacteria showed that all surface had the antibacterial activity. However, of all the sample, PLA nanofiber containing crude mormodica oil and ozonated one had higher antibacterial activity against to *S.aureus*. On the other hand all sample did not show antibacterial activity against to *E.coli* (Table 3). This situation could be explained by lack of the amount of oil in the nanofiber. Because it was seen that antibacterial activity of the oil increased in tandem with the escalating of the oil amount according to the disc diffusion method. Thus, the more oil is contained in the nanofiber, the more antibacterial activity is observed.

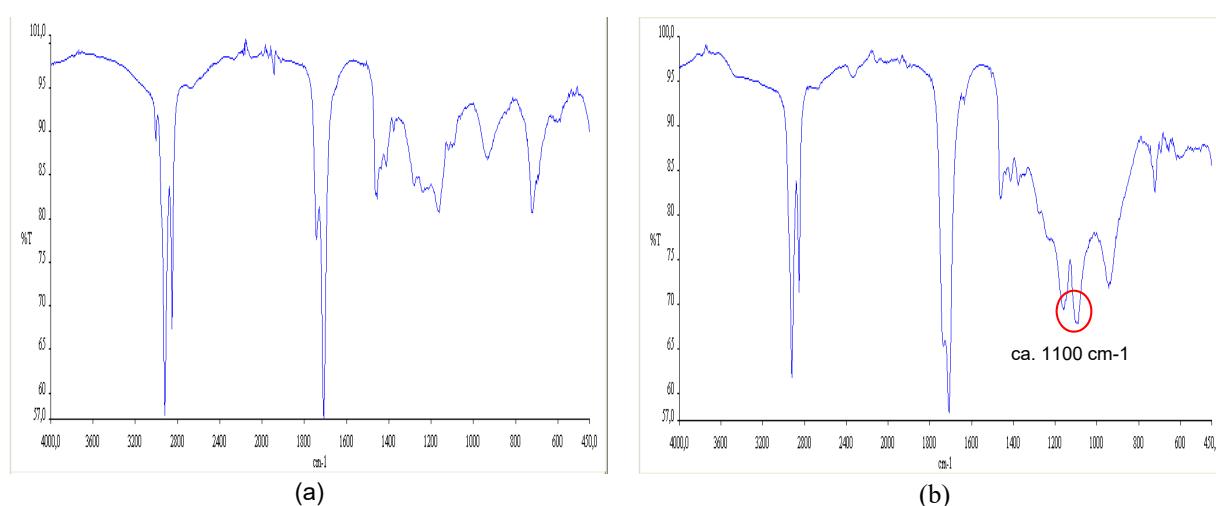


Figure 3. FT-IR spectrum of the oil a.mormodica oil b. ozonated mormodica oil.

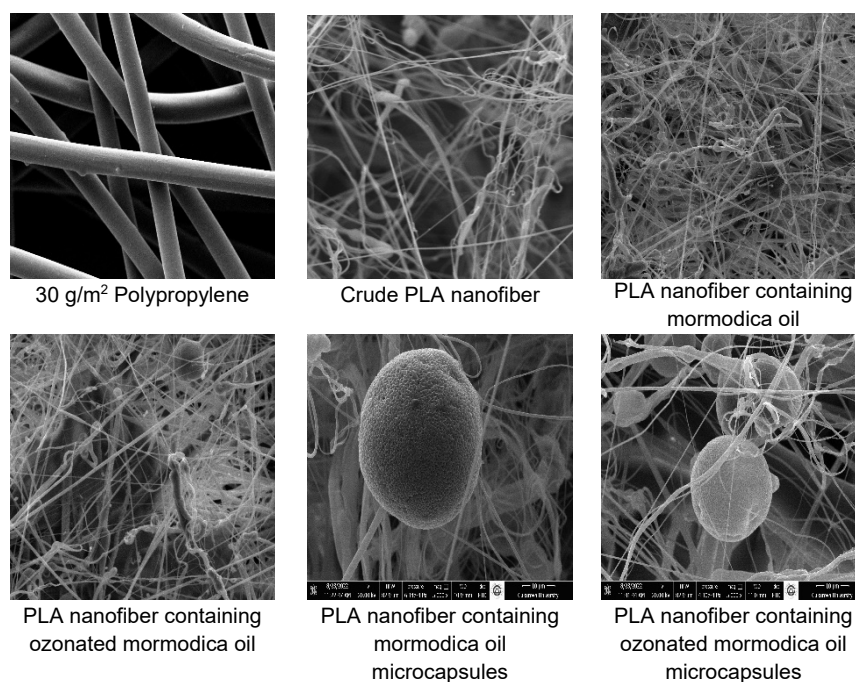


Figure 4. SEM images of the nanofiber.

Table 3. Antibacterial activity of the non-woven and nanofiber surfaces.

Sample	Bacteria reduction/proliferation (%)	
	<i>S.aureus</i>	<i>E.coli</i>
Polypropylene ground non-woven surface	-88,25	-11,48
PLA nanofiber	-83,51	-12,41
PLA nanofiber containing mormodica oil	-99,37	-11,11
PLA nanofiber containing ozonated mormodica oil	-100,00	-40,74
PLA nanofiber containing mormodica oil micro capsules	-81,40	-11,67
PLA nanofiber containing ozonated mormodica oil micro capsules	-98,72	-10,19

CONCLUSIONS

In the scope of this study, ozonated oil showing antibacterial activity were obtained. After that, crude and ozonated mormodica oil were microencapsulated via simple coacervation successfully. PLA nanofibers were spun by solution blowing spinning. Moreover, PLA nanofiber containing oil and microcapsules were spun successfully but some beads formation in the surfaces was observed. Nevertheless, All surface show antibacterial activity against to *S.aureus* while they did not show antibacterial activity against to *E.coli*. For further studies, it is thought that fiber formation parameters of the solution blowing spinning for PLA polymer will investigate deeply and will try to obtain antibacterial activity of the surfaces against to gram negative bacteria (*E.coli*).

Acknowledgements: This study (5220045) is funded by The Scientific and Technological Research Council of Turkey, Republic of Turkey (TUBITAK). We would like to thank warmly TUBITAK for supporting this study.

REFERENCES

- Beşen, B. S., Güneşoğlu, C., Balci, O., & Sütçü, K. (2016). Antibacterial Finishing of 100% Cotton Fabric with β -Cyclodextrin-Ozonated Olive Oil Inclusion Complex. *AATCC Journal of Research*, 3(6), 12–18. <https://doi.org/10.14504/ajr.3.6.3>
- Beşen, B. S., Güneşoğlu, C., Balci, O., Sütçü, K., Balci, O., Güneşoğlu, C., İrem Tatlı, I. (2016). Obtaining medical textiles including microcapsules of the ozonated vegetable oils. *AATCC Journal of Research*, 3(6), 12–18. <https://doi.org/10.1007/s12221-017-1212-8>
- Sancar BEŞEN, B., Balci, O., Orhan, M., Güneşoğlu, C., İrem TATLI, İ., Sütçü İmam Üniversitesi, K., ... Botanik Anabilim Dalı, F. (2015). Ozonlanmış Yağlar ile İşlem Uygulanmış Dokusuz Yüzeylerin Antibakteriyel Etkinliklerinin İncelenmesi An Investigation on Antibacterial Activities of Nonwovens Treated with Ozonated Oils. *Journal of Textiles and Engineer*, 22(100), 25. <https://doi.org/10.7216/1300759920152210003>
- Öcal, E., (2013), Ozon Yağının Kapsül Formasyonu Üzerindeki Etkisi. Uzmanlık Tezi. Hacettepe Üniversitesi Tıp Fakültesi. Ankara. s.14
- Karthikeyan, M., Ramachandran, T., Shanmugasundaram, O.L., (2014), "Synthesis, Characterization, and Development of Thermally Enhanced Cotton Fabric Using Nanoencapsulated Phase Change Materials Containing Paraffin Wax", *The Journal of The Textile Institute*, <https://doi.org/10.1080/00405000.2014.886368>
- Özyıldız, F., Karagönüllü, S., Basal, G., Uzel, A., Bayraktar, O., (2012), "Micro-encapsulation of Ozonated Red Pepper Seed Oil with Antimicrobial Activity and Application to Nonwoven Fabric", *The Society for Applied*

- Microbiology, *Letters in Applied Microbiology*, vol.56, pp.168-179.
<https://doi.org/10.1111/lam.12028>
7. Medeiros, E. S., Glenn, G. M., Klamczynski, A. P., Orts, W. J., & Mattoso, L. H. C. (2009). Solution blow spinning: A new method to produce micro- and nanofibers from polymer solutions. *Journal of Applied Polymer Science*, 113(4), 2322–2330.
<https://doi.org/10.1002/app.30275>
 8. Calisir, M. D., & Kiliç, A. (2020). A comparative study on SiO₂ nanofiber production via two novel non-electrospinning methods: Centrifugal spinning vs solution blowing. *Materials Letters*, 258, 126751.
<https://doi.org/10.1016/j.matlet.2019.126751>
 9. Polat, Y., Calisir, M., Gungor, M., Sagirli, M. N., Atakan, R., Akgul, Y., ... Kiliç, A. (2019). Solution blown nanofibrous air filters modified with glass microparticles. *Journal of Industrial Textiles*, 1–14.
<https://doi.org/10.1177/1528083719888674>
 10. Shi, L., Zhuang, X., Tao, X., Cheng, B., & Kang, W. (2013). Solution blowing nylon 6 nanofiber mats for air filtration. *Fibers and Polymers*, 14(9), 1485–1490.
<https://doi.org/10.1007/s12221-013-1485-5>
 11. Sinha-Ray, S., Sinha-Ray, S., Yarin, A. L., & Pourdeyhimi, B. (2015). Application of solution-blown 20-50nm nanofibers in filtration of nanoparticles: The efficient van der Waals collectors. *Journal of Membrane Science*, 485, 132–150.
<https://doi.org/10.1016/j.memsci.2015.02.026>
 12. Liu, Y., Zhang, G., Zhuang, X., Li, S., Shi, L., Kang, W., ... Xu, X. (2019). Solution blown nylon 6 nanofibrous membrane as scaffold for nanofiltration. *Polymers*, 11(2), 1–15.
<https://doi.org/10.3390/POLYM11020364>