SERTIFIKAT

No : 51331/UN38.3/DL.01.02/2022

Diberikan kepada :

Sania Nur Faiza

Atas partisipasinya sebagai :

Pemakalah

Dengan judul:

Fabrication of Nanofiber Membranes: PVA-SiO2 Prepared by Electrospinning Method for Lithium Battery Separator

Dalam Seminar Nasional Fisika 2022 dengan tema:

"Rekognisi Hasil Inovasi Fisika dan Aplikasinya Menuju Era Keterbukaan Informasi Ilmiah"

Diselenggarakan oleh Jurusan Fisika FMIPA Unesa pada Tanggal 27 Agustus 2022

Dekan FMIPA

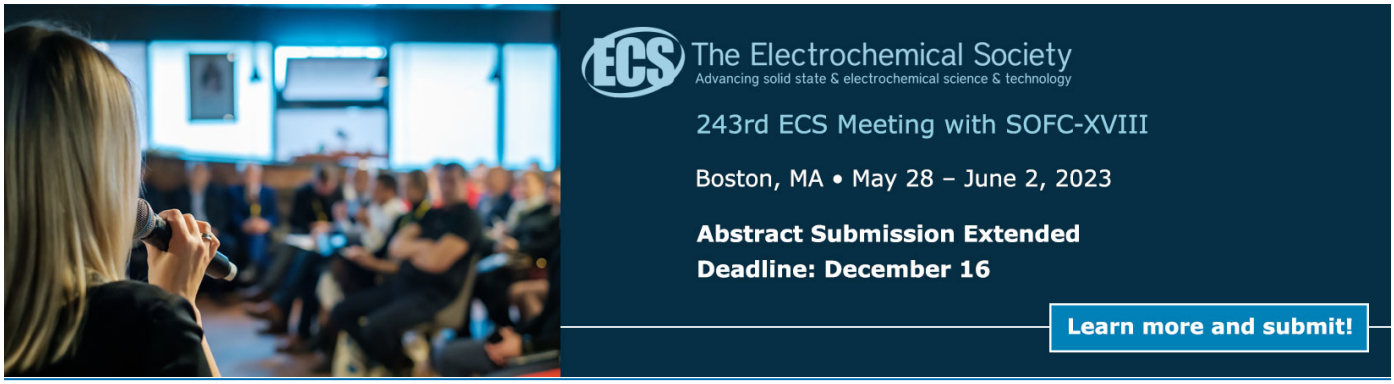
Prof. Dr. Madlazim, M.Si.
NIP 196511051991031012

Ketua Panitia

Dr. Binar Kurnia Prahani, M.Pd.
NIP 199005132019031012

Terpublikasi di:
IOP science Publishing JPFA-UNESA

Effectiveness of Polyvinyl Alcohol Nanofiber Composites as Anti-Bacterial Materials in Wound Dressing



Journal of Physics: Conference Series **2392** (2022) 012018 doi:10.1088/1742-6596/2392/1/012018

Effectiveness of Polyvinyl Alcohol Nanofiber Composites as Anti-Bacterial Materials in Wound Dressing

D H Kusumawati*, Munasir, L Rohmawati, P Uzalia, M D Layli, A B Rahanti, and I Yuliani

Department of Physics, Universitas Negeri Surabaya, Indonesia

***E-mail:** diahkusumawati@unesa.ac.id

Abstract. Closing the wound using a wound dressing can speed up the wound healing process. The ideal wound dressing has the characteristics of being able to maintain the humidity of the environment around the wound and prevent the entry of microorganisms. In addition, wound dressings also do not cause a residual effect on the skin when removed, which can cause an irritating effect on the skin. This effect can be minimized by using a very fine fibrous wound dressing, in this case using a very small fiber (nanofiber). Materials that meet the criteria for wound dressings include polyvinyl alcohol (PVA). PVA composite nanofibers can be fabricated using the Electrospinning method. The nanofiber composites used include PVA/PVP/Chitosan, PVA/ZnO, PVA/Gelatin and PVA/Chitosan. Where the composite material has the ability to absorb, and the fiber diameter is in the range of 130-230 nm. Based on the anti-bacterial characterization, PVA composite effectively blocks the entry of microorganisms for approximately 3 - 4 hours, with efficiency ranging from 47.6% - 95.7%. The lowest efficiency was found in PVA/Gelatin composites and the highest in PVA/ZnO.

1. Introduction

Nanotechnology products that are currently being developed include nanofibers which have applications in various fields [1]. Nanofibers can be fabricated using the Electrospinning method, where this method is very easy to do with the results of relatively small diameter nanofibers on the nanoscale. Applications of nanofibers in biophysics (medical physics) include the use of nanofibers in accelerating the wound healing process on the outer surface of the human body [2].

One of the efforts in the wound healing process is the use of wound dressings which are expected to function to close the wound and accelerate the wound healing process. The ideal wound dressing must be able to maintain the humidity of the environment around the wound, absorb exudate and prevent the entry of microorganisms along with gas exchange that allows the skin in the wound area to obtain oxygen, thereby accelerating the wound drying process. The wound dressing must also be non-toxic, non-allergenic and antimicrobial to speed up the wound healing process. One of the weaknesses of current gauze dressings is the relatively large pressure when used and requires a pull when released, and this has the potential to leave a residue from the gauze, which can form granulomas and cause skin irritation. To overcome this, it is necessary to use finer gauze fibers by applying nanotechnology in making nanofibers for wound dressings using the Electrospinning method. The materials used in this

case are Polyvinyl Alcohol (PVA), chitosan, gelatin and ZnO. PVA is non-toxic, biodegradable and has good biocompatibility and mechanical properties. Meanwhile, chitosan, gelatin and ZnO have anti-bacterial, anti-fungal, analgesic, non-toxicity, biodegradable and biocompatibility characteristics and have the ability to effectively deliver extrinsic antimicrobial compounds to the infected wound area.

Electrospinning is one of the fastest-growing polymer-based nanofiber fabrication methods. Electrospinning is an "electric spinning" technique that produces nanometer-sized fibers, by utilizing the electrostatic force due to the potential difference between the sample and the substrate (drum collector). Electrospinning can produce fibers with diameters ranging from nanometers to micrometers [1] and has a high surface area [2]. The resulting nanofibers can be applied as tissue engineering [3], drug delivery in anticancer [4], cosmetics [5], and as wound dressings [6-8]. This nanofiber membrane structure has good wound fluid absorption ability, vapor transmission rate, and drug or addictive transfer rate [2]. The polymer material used must have good chemical, mechanical, temperature stability, biocompatibility, electrical conductivity, and electrical activity [9]. PVA is a water-soluble polymer and has excellent chemical resistance, biocompatibility, physical properties, biodegradability, and non-toxic [3].

PVA is a water-soluble polymer and has excellent chemical resistance, biocompatibility, physical properties, biodegradability, and non-toxic [3]. However, PVA has a tendency to decrease in viscosity with increasing temperature [10]. Polyvinyl alcohol is a non-toxic transparent synthetic polymer with high biocompatibility and biodegradability. It is also soluble in water, highly polar, and forms many positive interactions through the hydroxyl group [11]. This polymer is non-toxic and, when in contact with the skin, does not cause damage. Therefore it is used as a consideration in wound dressing applications and is easily mixed with various materials [12]. PVP is an amorphous synthetic polymer with a high affinity for water and good adhesion [13]. It is easily soluble in water, physiologically compatible, non-toxic, chemically inert, temperature resistant, pH stable, biodegradable and colorless polymer [2]. This PVP material can be applied in the biomedical field as a new wound dressing material that provides and maintains a moist environment to prevent scab formation and dehydration of the wound bed [14]. Chitosan is an ingredient that can promote wound healing, exhibits anti-bacterial activity and good absorption, as well as being able to produce fibers that have a porous structure and high surface area [15]. Chitosan is one of the biomaterials that can be made using the electrospinning method.

Chitosan is a polycationic polysaccharide which has the characteristics of high biodegradability and biocompatibility [13]. Chitosan also has advantages such as antimicrobials to prevent and treat wound infections [14]. Gelatin is biocompatible, non-cytotoxic, and biodegradable when used for clinical applications [16]. In addition, gelatin can increase adhesion, contributing to faster wound healing [17]. ZnO to further stabilize the polyvinyl alcohol (PVA) polymer solution [18]. Nano-sized ZnO particles have stronger antimicrobial activity when compared to larger particles due to their large surface-volume ratio, which allows better interaction with bacteria [19]. The application of ZnO nanoparticles as an anti-bacterial agent depends on the process control properties such as particle size, size distribution, shape, surface area, and dispersion [20]. The characteristics of PVA and its composite forming materials, (PVP, Chitosan, Gelatin and ZnO), meet the criteria as a material for making wound dressings. So, a material that is good and in accordance with the standard of the wound covering material will be obtained. The purpose of this research is to synthesize PVA/Chitosan, PVA/PVP/Chitosan, PVA/Gelatin and PVA/ZnO nanofibers and characterize them as antimicrobial wound dressings that are effective and efficient in assisting the wound care process that does not cause new infection effects in the area around the wound.

2. Methods

The material used in making nanofibers is polyvinyl alcohol dissolved in distilled water with a concentration of 10%. As materials for making nanofiber composites, gelatin, chitosan, ZnO and PVP

are used. The material is dissolved with distilled water and mixed with a PVA solution of a certain composition according to the characteristics of the base material. The tools used in making the solution include a beaker glass, digital balance, stirrer and electrospinning (Nachriebe 601). The solution that is ready to be spun is put into the spet as much as approximately 10 ml. While the characterization tools used are FTIR, SEM and anti-bacterial test (International Standard ISO22196).

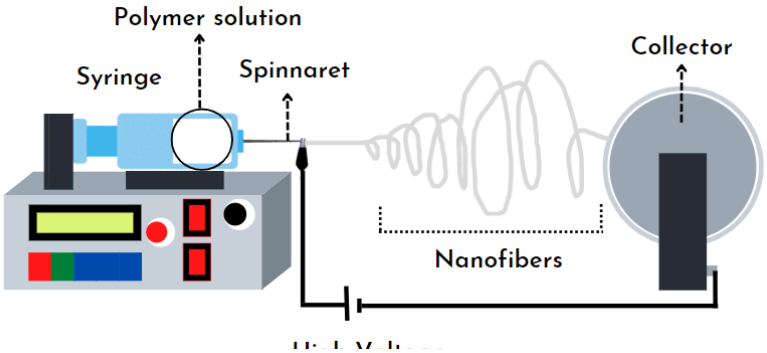
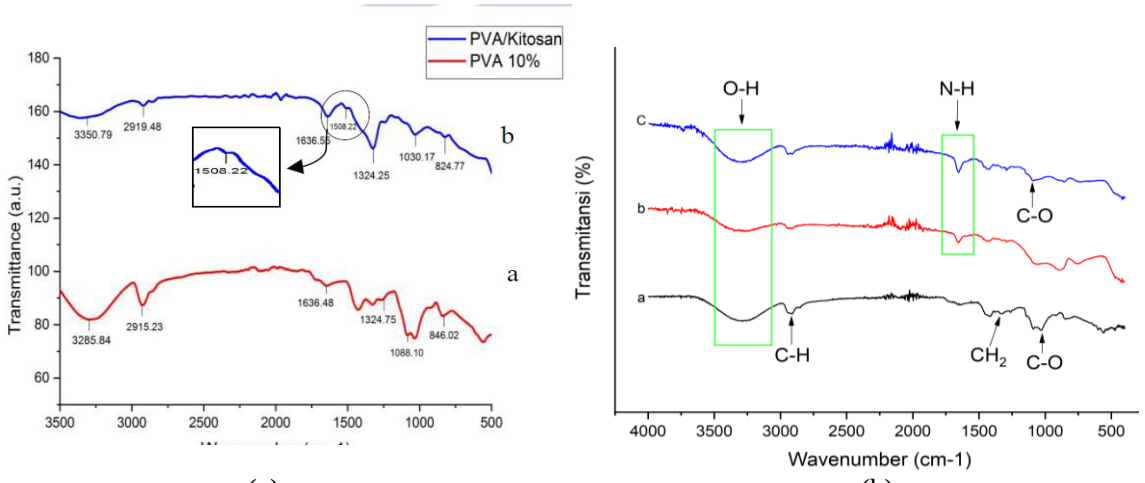


Figure 1. Electrospinning process

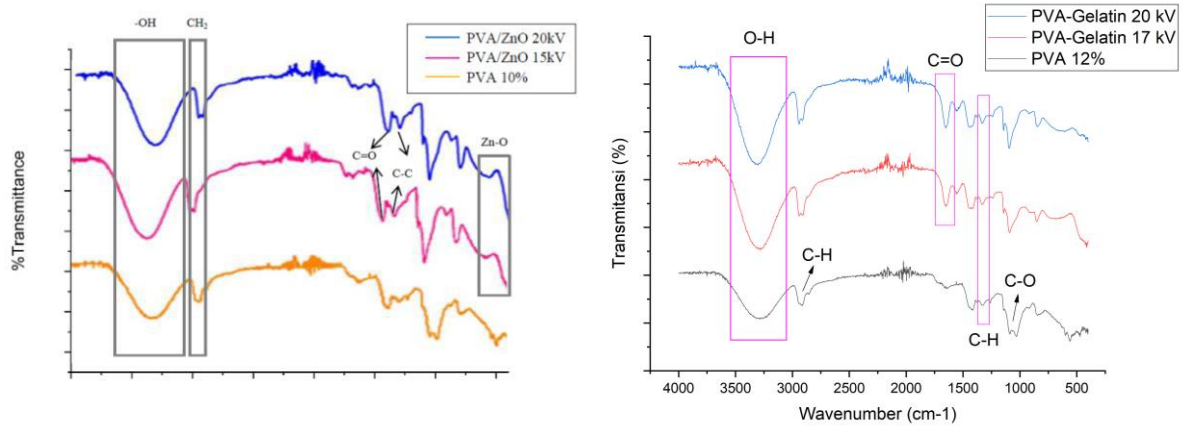
Making PVA solution using the wet mixing method, the nanofiber is produced by electrospinning (Nachriebe 601). Mixing the PVA solution with other ingredients (gelatin, PVP, Chitosan and ZnO) with the wet mixing method, stirred at room temperature for approximately 2-4 hours, until a homogeneous solution is produced. The electric spinning process is carried out with varying voltages between 12-21 kV, the distance from the spet to the drum collector is 12-18 cm, with a spinning time of 1-3 hours. The electrically spun nanofibers were characterized using FTIR, to determine the functional groups formed. Then SEM determines the morphology of the fiber and the diameter of the resulting fiber. Furthermore, an anti-bacterial test was carried out according to the International Standard ISO22196 with Staphylococcus aureus bacteria.

3. Results and Discussion

The PVA solution produced after adding other ingredients (gelatin, PVP, Chitosan and ZnO) was continued into the spinning process. There are four types of solutions, namely PVA/Chitosan, PVA/PVP/Chitosan, PVA/Gelatin and PVA/ZnO. Each solution is made into nanofibers using an electric spinning process. The resulting nanofibers were characterized using FTIR to determine the functional groups formed. The results of the FTIR are shown in Figure 2.



(a) (b)



(c) (d)

Figure 2. FT-IR results for (a) PVA/Chitosan, (b) PVA/PVP/Chitosan, (c) PVA/ZnO, and (d) PVA/Gelatin.

Figure 2 shows that in each solution it has been identified that the functional group of the nanofiber is the functional group of the basic ingredients that make it up. Figure 2(a) is the result of PVA/Chitosan, where the presence of the N-H amide II functional group was identified as a functional group that characterizes chitosan at a wave number of 1608 cm^{-1}[21]. In Figure 2(b), there are functional groups that identify PVP and chitosan, where PVP is identified as having the same group as PVA, and chitosan is identified with an N-H group at a wave number of 1617 cm^{-1}. In the PVA/ZnO nanofiber (Figure 2(c)), there is a Z-O functional group in the wave number 400-600 cm^{-1} [22,23]. Meanwhile, the PVA/Gelatin nanofibers (Figure 2(d)) have almost the same functional groups, so it can be identified that the higher peaks that appear in several functional groups reinforce each other between the two materials, namely the C=O stretching functional group with a number wave 1650-1735 cm^{-1}[24].

Table 1. FTIR characterized functional group

Functional Group	Wave number (cm^{-1})	Material Identification	Reference
N-H amide II	1608-1617	Chitosan	[21]
N-H			
Z-O	400-600	ZnO	[22,23]
C=O	1650-1735	Gelatin	[24]

Based on the results of FTIR characterization, it can be ascertained that the nanofiber formed is already a nanofiber containing added materials (not only PVA), namely chitosan, PVP, ZnO and gelatin. To determine the physical characteristics of the fiber formed, the diameter and morphology of the fiber were measured using SEM. Wound dressing to be made must have a homogeneous and smooth surface. Thus, the fiber with a very small diameter can be used as a wound cover that does not cause skin tissue damage when removed. The results of the SEM characterization are shown in Figure 3.

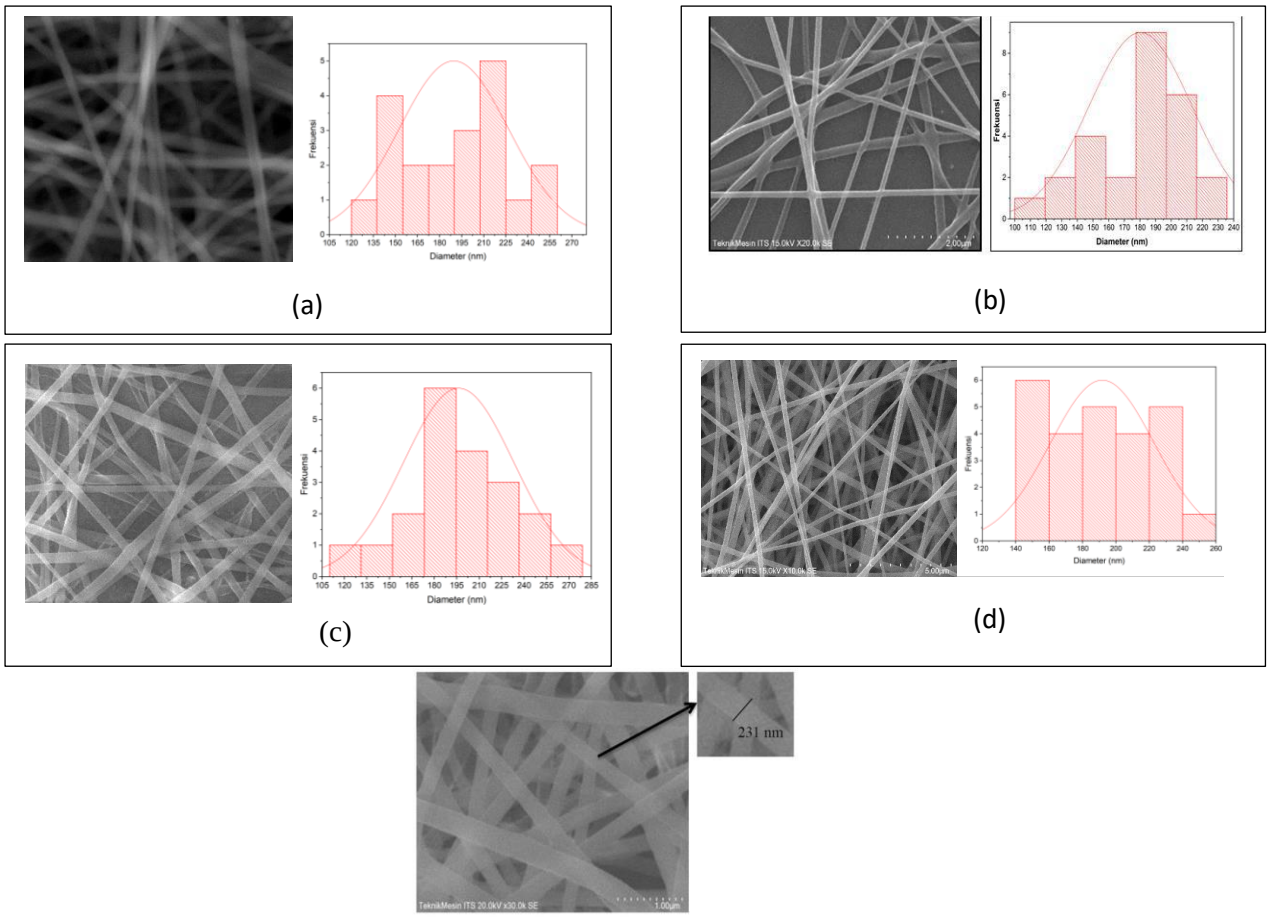


Figure 3. Result SEM from (a) nanofiber PVA, (b) nanofiber PVA/Gelatin, (c) nanofiber PVA/PVP/Chitosan, (d) nanofiber PVA/Chitosan, and (e) nanofiber PVA/ZnO

The results of SEM characterization, the morphology of the resulting nanofibers showed the best homogeneity in PVA/Chitosan and PVA/ZnO, where it was seen that the fibers formed were denser and the diameter of the fibers produced was almost the same size. In PVA/Gelatin, it can be seen that the fiber formed is very rare even though the diameter of the fiber is smaller and there are fibers that turn into beads when attached to other fibers. PVA/PVP/chitosan is almost the same as PVA/Gelatin, but the fibers produced are denser than PVA/chitosan. Beads that appear more, and between the fibers seen a lot of sticking together.

The diameter of the PVA fiber before adding other materials produced varies from 120-255 nm, where the diameter of the fiber used as a wound dressing is 100-300 nm [25]. However, PVA does not have anti-bacterial characteristics, so it is necessary to add other ingredients. Based on the SEM results, after PVA was given an anti-bacterial additive, the fiber diameter obtained was still in the range of 100-285 nm, meaning that it still met the criteria as a wound dressing. The diameter is bigger because there are some fibers that stick together which causes changes in the shape and dimensions of the fiber to widen.

Testing wound dressing as an anti-bacterial using Staphylococcus aureus bacteria which is a gram-positive bacteria that breeds in wounds, especially wounds on the outer skin tissue. The test method used conforms to the International Standard ISO22196 regarding specifying a method of evaluating the anti-bacterial activity of antibacterial-treated plastics, and other non-porous, surfaces of products (including intermediate products)[26]. This test method used the initial number of bacteria as a control,

namely 723 colonies. The test is done by varying the time of using wound dressing for 1-5 hours with an interval of 1 hour checking the number of bacterial colonies in the wound dressing.

Table 2. Anti-bacterial test results on wound dressing

Time (hour)	PVA/Chitosan		PVA/PVP/Chitosan		PVA/Gelatin		PVA/ZnO	
	Colony	Efficiency (%)	Colony	Efficiency (%)	Colony	Efficiency (%)	Colony	Efficiency (%)
1 hour	278	61.6	715	1.1	544	24.8	253	65.0
2 hour	335	53.7	713	1.4	379	47.6	172	76.2
3 hour	73	89.9	312	56.8	450	37.8	31	95.7
4 hour	184	74.6	632	12.6	517	28.5	213	70.5
5 hour	229	68.3	689	4.7	550	23.9	218	69.8

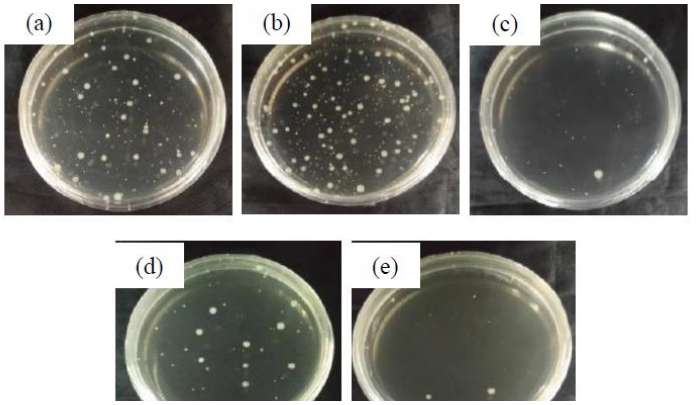


Figure 4. Image of a colony on PVA/ZnO wound dressing anti-bacterial test

From the results of the anti-bacterial test, PVA-based wound dressings have met the criteria as wound dressings if the efficiency of bacterial absorption reaches 50% and the usage time is 3-4 hours. [7,16,18]. Wound dressing which has the highest efficiency is PVA/ZnO reaching 95.7% with a usage time of 5 hours still able to absorb with an efficiency of around 70%. Likewise, with PVA/Chitosan, the efficiency reached 89.9% and within 5 hours was still able to absorb with an efficiency of 68%. It is different with PVA/PVP/Chitosan which only has an efficiency of 56.8% after 3 hours of use, and will decrease drastically when used for more than 3 hours. Meanwhile, the highest efficiency of PVA/Gelatin only reached 47.6% for 2 hours of use and continued to decrease with increasing usage time. When viewed from the characteristics of the nanofibers formed, the low efficiency of PVA/Gelatin and PVA/PVP/Chitosan is due to the presence of fiber parts that stick together and form beads that hinder the absorption process of bacteria entering the wound dressing [8,17].

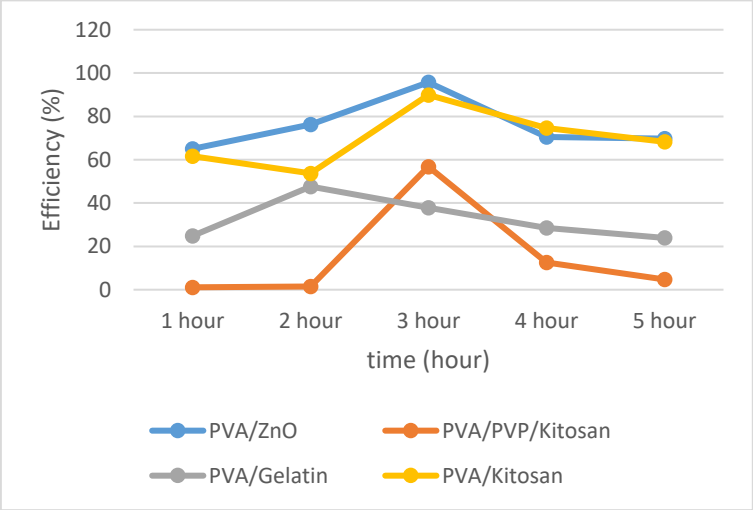


Figure 5. Anti-bacterial absorption efficiency composite of PVA

4. Conclusion

Wound dressings can be made from polymer materials, namely PVA, combined with other materials that help absorb bacteria that arise for types of wounds on the skin tissue. The efficiency of wound dressing against Staphylococcus aureus bacteria, the best is wound dressing made from PVA/Chitosan and PVA/ZnO, where the efficiency reaches 90% and is still able to absorb above 50% for a period of 5 hours.

Acknowledgements

We would like to thank all those who supported this research, especially the Universitas Negeri Surabaya, which has funded this research, through PNBP funds. I also thank the research team, both lecturers and students, for their cooperation and support during the research.

References

[1] Faridi-Majidi R, Nezafati N, Pazouki M, and Hesaraki S 2017 *Nanomed. J.* **4** 107.
[2] Asawahame C, Sutjarittangtham K, Eitssayeam S, Tragoolpua Y, Sirithunyalug B, and Sirithunyalug J 2015 *AAPS PharmSciTech* **16** 182.
[3] Subramanian UM, Kumar SV, Nagiah N, and Sivagnanam UT 2014 *Int. J. Polym. Mater. Polym. Biomater.* **63** 476.
[4] Bhusnure O G, Gholve S B, Giram P S, Gaikwad A V, Udumansha U, Mani G, and Tae J H 2021 *J. Drug Deliv. Sci. Technol.* **64** 102654.
[5] Laosirisathian N, Saenjum C, Sirithunyalug J, Eitssayem S, Chaiyana W, and Sirithunyalug B 2021 *Fibers Polym.* **22** 36.
[6] Alipour R, Khorshidi A, Shojaei AF, Mashayekhi F, and Moghaddam M J M 2019 *Polym. Test.* **79** 106022.
[7] Gökmeşe F, Uslu I, and Aytimur A 2013 *Polym. Plast. Technol. Eng.* **52** 1259.
[8] Rosli N and Setu S A 2018 *eProceedings Chem.* **3** 152.
[9] Rihova M, Ince A E, Cicmancova V, Hromadko L, Castkova K, Pavlinak D, Vojtova L, and Macak J M 2021 *J. Appl. Polym. Sci.* **138** 49975.
[10] Hashmi M, Ullah S, Ullah A, Akmal M, Saito Y, Hussain N, Ren X, and Kim I S 2020 *Polymers* **12** 2524.
[11] Barkoula N M, Alcock B, Cabrera N O, and Peijs T 2017 *Polym. Polym. Compos.* **16** 101.
[12] Baghaie S, Khorasani M T, Zarrabi A, and Moshtaghian J 2017 *J. Biomater. Sci. Polym. Ed.* **28** 2220.
[13] Rahmani F, Ziyadi H, Baghali M, Luo H, and Ramakrishna S 2021 *Appl. Sci.* **11** 2779.

[14] Aytimur A and Uslu I 2014 *Polym. Plast. Technol. Eng.* **53** 655.
[15] Zhao Y, Qiu Y, Wang H, Chen Y, Jin S, and Chen S 2016 *Int. J. Polym. Sci.* **2016** 4672839.
[16] Tomitaka A, Jo, J, Aoki I, and Tabata Y 2014 *Inflamm. Regen.* **34** 45.
[17] Sengor M, Ozgun A, Gunduz O, and Altintas S 2020 *Mater. Lett.* **263** 127233.
[18] Jamnongkan T, Kaewpirom S, Wattanakornsiri A, and Mongkholrattanasit R 2018 *Eng. Mater* **759** 81.
[19] Siddiqi K S, Rahman A, Tajuddin, and Husen A 2018 *Nanoscale Res. Lett.* **13** 141.
[20] Dimapilis E A S, Hsu C S, Mendoza R M O, and Lu M C 2018 *Sustain. Environ. Res.* **28** 47.
[21] Das P, Ojah N, Kandimalla R, Mohan K, Gogoi D, Dolui S K, and Choudhury A J 2018 *Int. J. Biol. Macromol.* **114** 1026.
[22] Rasouli R, Barhoum A, Bechelany M, and Dufresne A 2019 *Macromol. Biosci.* **19** 1800256.
[23] Aderibigbe B A and Buyana B 2018 *Pharm.* **10** 42.
[24] Yang Z, Peng H, Wang W, and Liu T 2010 *J. Appl. Polym. Sci.* **116** 2658.
[25] Vilamova Z, Konvickova Z, Mikes P, Holisova V, Mancik P, Dobrocka E, Kratosova G, and Seidlerova J 2019 *Sci. Rep.* **9** 15520.
[26] ISO 2011 *ISO 22196:2011 Measurement of antibacterial activity on plastics and other non-porous surfaces* https://www.iso.org/standard/54431.html.