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Polyvinyl-alcohol/polyvinyl-pyrrolidone membranes coated with violacein pigments as antibacterial agent for wound dressing application

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GRAPHICAL ABSTRACT

PVA/PVP-violacein membrane

ABSTRACT

Polyvinyl-alcohol (PVA) and polyvinyl-pyrrolidone (PVP) are among common polymers used in producing wound dressing due to their non-toxicity, biodegradable, and hydrophilicity properties. Two methods were used to produce the PVA/PVP membranes which are casting and electrospinning. The membranes were dip-coated into violacein pigment as an antibacterial agent. The results from attenuated total reflectance Fourier transform infrared (ATR-FTIR) show that, using electrospinning, the PVA/PVP membrane was chemically bonded and crosslinked while by casting the polymers form physical bonding. Meanwhile, the tensile strength analysis gave a modulus elasticity value of electrospun membrane (4.2662 N), casted membrane (2.8029 Nm^{-2}), electrospun-violacein membrane (2.0959 N), and casted-violacein membrane (0.7287 N). Field emission sanning electron microscopy (FESEM) shows where electrospun membranes have fibres with beads and swollen after dipped into violacein pigment. Casted membranes have no fibre and have no difference after dipped into violacein pigment. In thermal analysis, an exothermic process and multistage decomposition occurred where all the membranes showed two degradation points. The release rate of the membranes increased with time where the constant concentrations of electrospun and casted membranes were 35.176 µg and 25.176 µg respectively. The antibacterial activity gave positive results in positive control of the electrospun PVA/PVP-violacein membrane where the inhibition region yield was 30.8%. In conclusion, this study shows that electrospun membranes give better results over casted membranes and the addition of antibacterial agents gives added values for the membranes to be applied as wound dressing.

Keywords: electrospinning, violacein, PVA, PVP, membrane

1. INTRODUCTION

There is a high demand in improving wound care products due to the surgical procedures every year. Wound dressing can be defined as a pad or compress that directly in contact with the wound either with or without medicine which will create a moist environment for faster healing rate [1]. Then, the blend of polyvinyl-alcohol (PVA) and polyvinyl-pyrrolidone (PVP) polymeric solutions will produce the desired wound dressing materials with enhance mechanical, degradation, and biocompatibility properties of membrane [4] due the properties of the polymers. Both, PVA and PVP are hydrophilic polymers. PVA is biocompatible and non-toxic which can be easily handled and has high water permeability. These excellent properties of PVA lead it to be used in various fields such as medical, cosmetic, pharmaceutical and packaging materials in industries [2]. Furthermore, PVP is also biocompatible and non-toxic polymer. PVP is readily soluble in water, elastic, transparent, flexible, impermeable for bacteria and will get attached to healthy skin but not wound as it will not hurt the wound. However, PVP properties are only suitable for topical environment wound dressing [3]. The most common used technique in fabrication is electrospinning technique as it can fabricate from micro to nano scale fibres using polymeric solution. The fabricated membranes have several advantages such as high surface area to volume ratio, tunable porosity and ability to manipulate the composition to produce the desired properties and functions [5]. Another method is casting of thin membrane as this method is easier to handle, safe cost and time. Violacein is a natural-occurring pigment that has anti-bacterial properties which is used in cosmetics, textiles, food, toys, and insecticides [7]. As this pigment contains bacterial agent, it will promote faster healing rate and prevent from bacteria attacking the wound.

The aim of this study is to fabricate and characterize membranes from a blended PVA/PVP polymer solution using two methods which are electrospinning and casting. Membranes were incorporated with violacein to improve the polymeric membrane wound dressing in terms of its antibacterial properties.

2. EXPERIMENTAL

The experiment was divided into three main stages. The first stage focused on producing the PVA/PVP membranes from two methods which are electrospinning and casting. In addition, the second stage was to incorporate the violacein pigment onto the membrane's surfaces. The last stage was the characterization of the PVA/PVP and PVA/PVP-violacein membranes from both methods. In order to produce PVA/PVP membranes, PVA (20 wt%) was dissolved in distilled water and stir with heat at temperature 124ºC for about an hour while PVP (30 wt%) was dissolved in ethanol. The two polymeric solution mixed in ratio of PVA:PVP; 100:0 and 75:25 until homogenous solution formed. In the second stage, the polymeric solution then electrospun at distance between the tip and collector is 20 cm, flow rate at 0.5 mL/h, 18-gauge needle used and voltage in the range of 20-25kV. Meanwhile, for casting method, the polymeric solution cast on smooth glass surface with certain thickness and dipped into acetone until membrane formed and peel off from the surface. Later, in stage three, the PVA/PVP and PVA/PVP-violacein membranes characterized to study their morphology by using FESEM, ATR-FTIR, tensile strength, thermal analysis, antibacterial activity test, and release rate.

3. RESULTS AND DISCUSSION

3.1 Morphology of membranes using FESEM

Figure 1 show the images from FESEM. The surface of electrospun membranes were smoother compared to cast membranes. Besides, fibre shape observed on electrospun membrane. However, electrospun membranes produced beads fibre which some parameters should be optimized to give better morphology. Both casted and electrospun membranes after dip-coated into violacein pigment become swollen. This is because the violacein solution contained ethanol, where the alcohol makes the membranes slightly expand as compared to membranes that not dip-coated with violacein. Hence, electrospinning produced membrane that formed by layering the crosslinked polymer and become thicker. For electrospinning technique, the parameters should be optimizing to produce less beads fibre. In this study, the lack of optimization caused the form of beads [8].

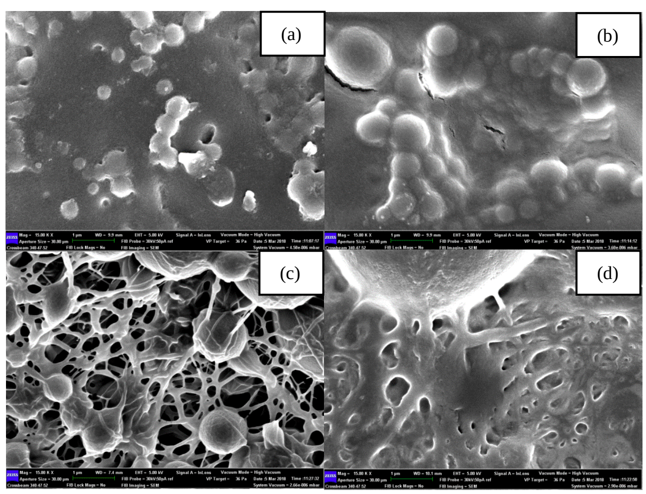


Figure 1 FESEM images of (a) cast PVA/PVP; (b) cast PVA/PVP-violacein; (c) electrospun PVA/PVP; and (d) electrospun PVA/PVP-violacein.

3.2 Tensile strength of membranes

The tensile strength of the tensile of the membranes is presented in the Figure 2. From the graphs, the highest elasticity value is electrospun membrane (4.2662 N), followed by casting membrane (2.8029 N) then, electrospun-violacein membrane (2.0959 N), and casting-violacein membrane (0.7287 N). This showed that the electrospun membrane is the stiffest compared to others. However, electrospun-violacein membrane was less stiff since the membrane was dip-coated into violacein and ethanol which make the membrane slightly brittle. However, the electrospun membranes show better mechanical properties compared to cast membranes as the fracture point of the membranes were higher. This is due to the well cross-linked polymerisation of the electrospun polymeric membrane [9].

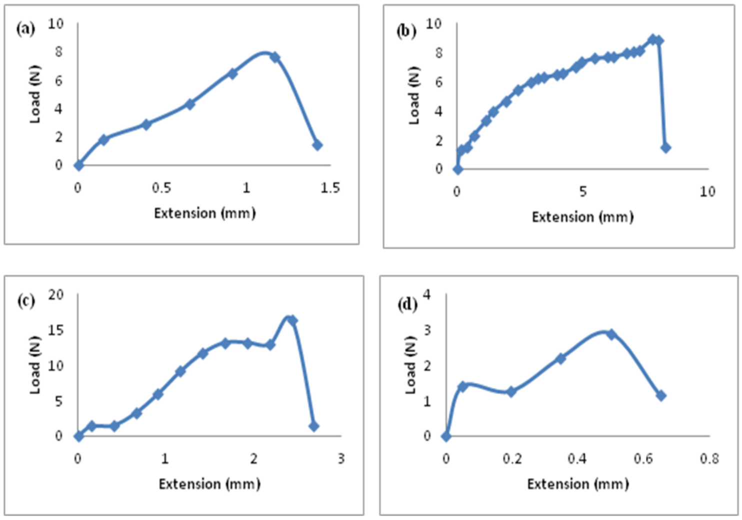


Figure 2 (a) The tensile strength of (a) cast PVA/PVP membrane; (b) cast PVA/PVP-violacein membrane; (c) electrospun PVA/PVP membrane; and (d) electrospun PVA/PVP-violacein membrane.

3.3 ATR-FTIR

Figure 3 shows the spectra of cast membranes. Casted membranes do not show much different because the compounds were physically bonded (PVA/PVP). There is broad O-H at 3264 cm-1 which represent the hydrogen bonding from hydroxyl group in PVA compound. C-H stretching at 2942 cm-1, 1089 cm-1 for C-O stretching also representing PVA compounds. For PVP, another C-H stretching peak at frequency of 2909 cm-1, along with that strong peak at 1652 cm-1 of C=O. There is no different between PVA/PVP and PVA/PVP-violacein membranes due to the method of incorporating the pigment onto the membranes. Hence, the spectra do not show any intense peaks for violacein. Figure 4 shows spectra for electrospun membrane. The huge difference of the peak intensity could be spotted between PVA membranes and PVA/PVP membranes where C=O stretching at 1648 cm-1 is sharp. Besides, at PVA/PVP-violacein spectra another peak at 1722 cm-1 may be represent to C=O of violacein [10].

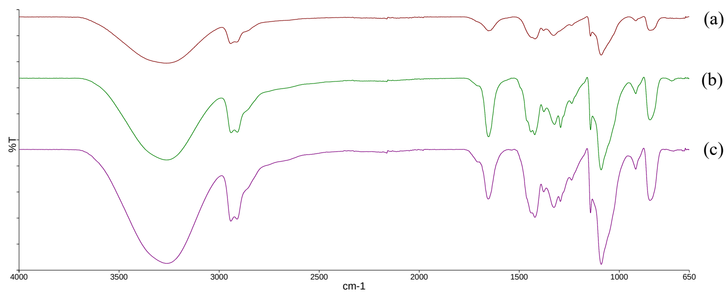


Figure 3 FTIR spectra of (a) PVA; (b) PVA/PVP; (c) PVA/PVP-violacein by using casting method.

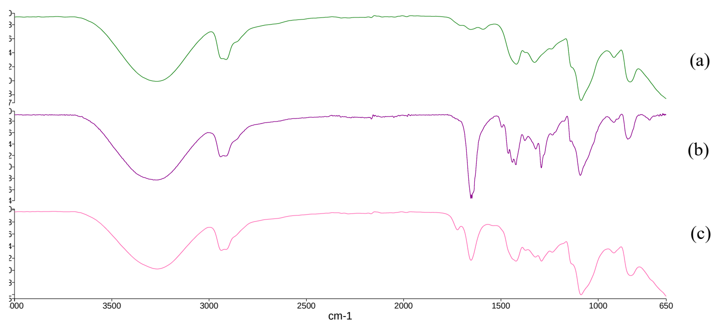
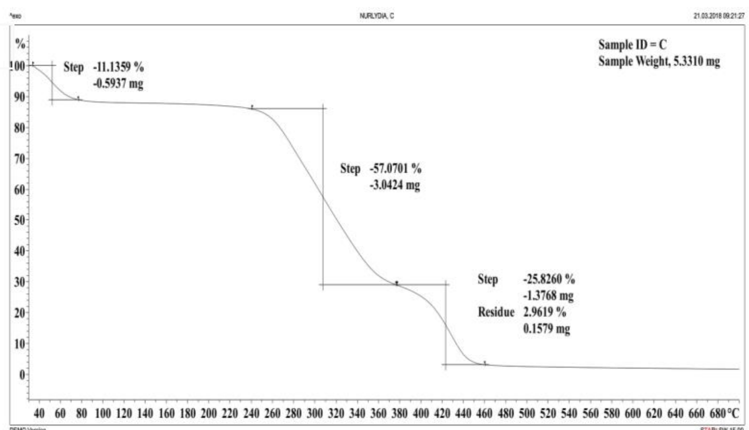


Figure 4 FTIR spectra of (a) PVA; (b) PVA/PVP; and (c) PVA/PVP-violacein membrane by using electrospinning method.

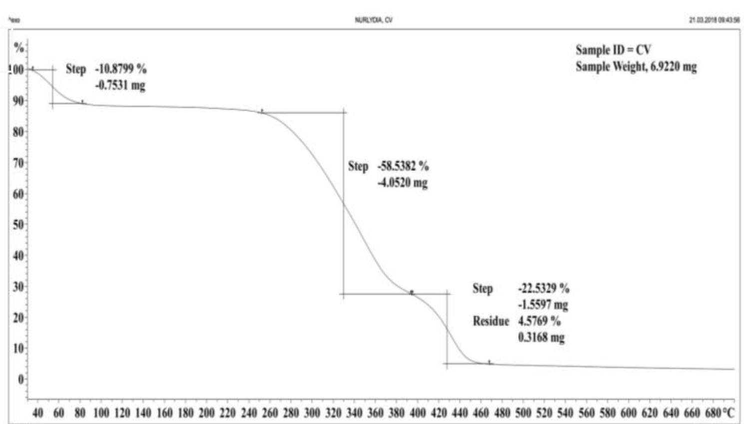
3.4 Thermal analysis of membranes

Figure 5 shows the graph of TGA of all membranes produced. All membranes display exothermic process and multi stage decomposition. At 40 ˚C, the process of removing moisture occurred. Based on TGA plot, there are two stages of degradation due to presence of by products at 300 ˚C and 420 ˚C [11]. However, at TGA-DSC plot at Figure 6 shows no glass transition, only melting point at 220 ˚C. Violacein do not occur at any peak since might be not crystallize and remain in amorphous state.

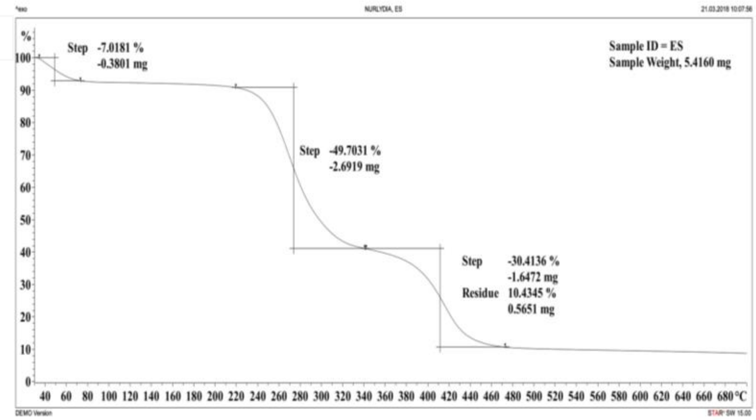
(a)



(b)



(c)



(d)

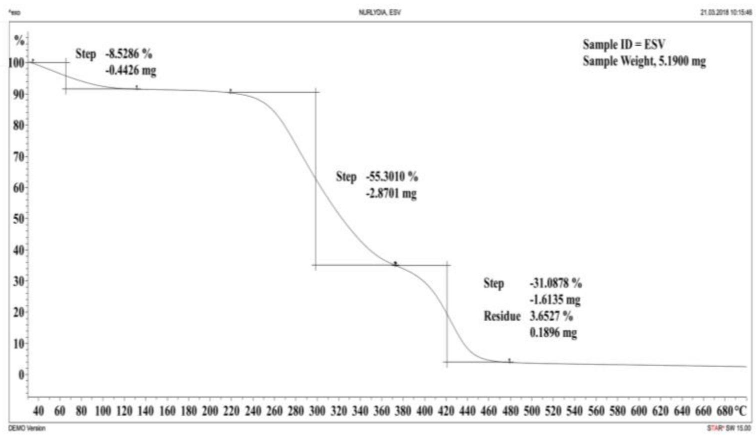
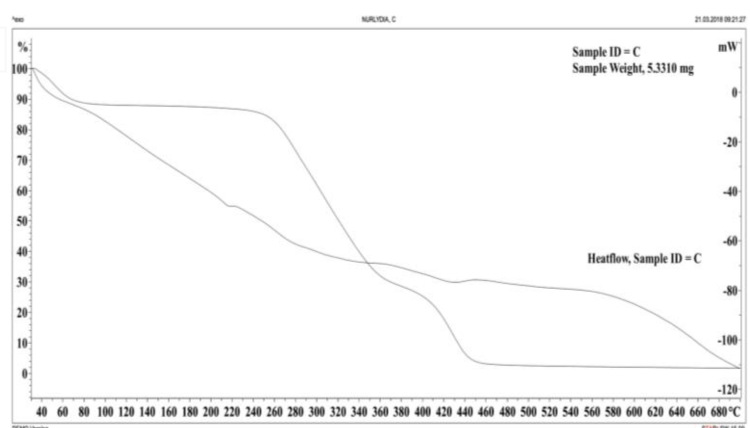


Figure 5 TGA plot of (a) casted PVA/PVP; (b) casted PVA/PVP-violacein; (c) electrospun PVA/PVP; and (d) electrospun PVA/PVP-violacein membranes.

(a)



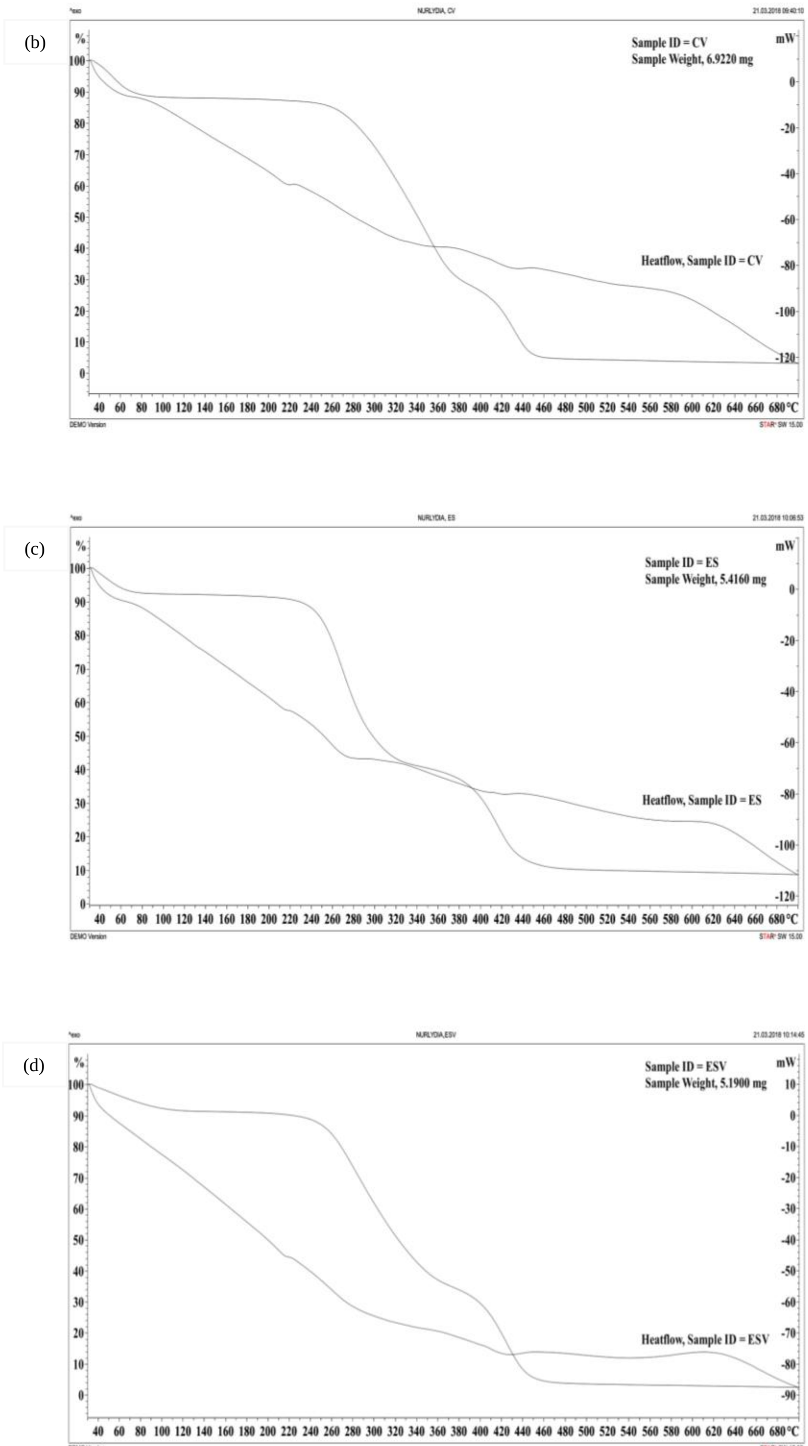


Figure 6 TGA-DSC plot of (a) casted PVA/PVP; (b) casted PVA/PVP-violacein; (c) electrospun PVA/PVP; and (d) electrospun PVA/PVP-violacein membranes.

3.2. Release rate of membranes

Based on Figure 7, casted membrane shows an increasing of absorbance from first one hour to three hours later and constant starting at fifth hours. Besides, electrospun membranes slightly increased after one hour and increased more at fifth hour. However, the amount of absorbed violacein pigment of electrospun membranes is greater than casted membranes as we can see the absorbance value. The molar absorptivity coefficient is 3.13x10 $^{-2}$ μg^{-1} cm^{-1} [12]. The electrospun membrane has higher absorptivity because of the fibre-shape. The layers of crosslinked polymers made the membrane thicker as compared to casted membrane where the membrane only forms a thin film-like membrane that cannot absorb more.

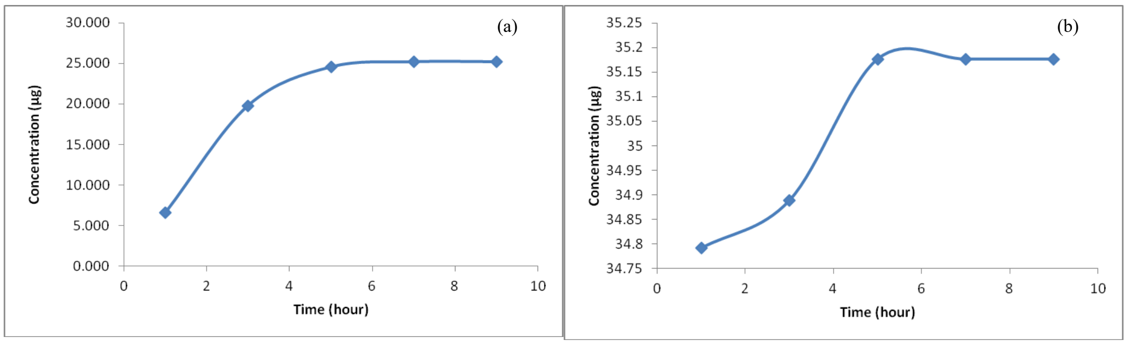


Figure 7 The release rate graph of (a) casted membranes and (b) electrospun membranes.

3.3 Antibacterial Activity

Disc-diffusion method have been used to study the antibacterial test of the membrane after dip-coated into violacein pigment. Based on Figure 8, at positive control, electrospun membranes that dip-coated with violacein pigment show a positive result where the inhibition region measured is 4 mm which is 30.8% of inhibition yield. Then, at negative control, all membranes show a negative result [13]. The presence of antibacterial activity is due to the layered of fibre-shape like polymers that can held violacein pigment more which could show a positive result. Apart from that, casted PVA/PVP-violacein membrane may have antibacterial properties but since the amounts of violacein that absorbed by the membrane are lower which make the inhibition region cannot be observed well.

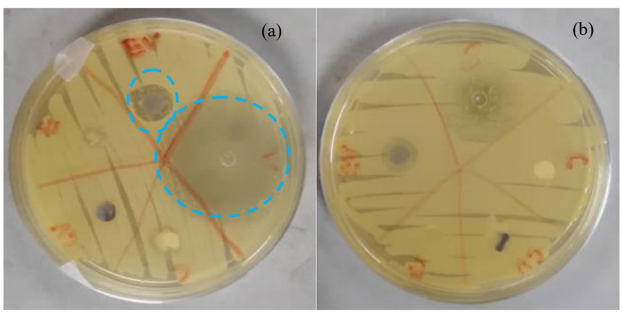


Figure 8 The antibacterial activity of the membranes at (a) positive control and (b) negative control.

4. CONCLUSION

This study shows that membranes produced by fabrication technique like electrospun give a better property for wound dressing application. Electrospinning incorporates the two polymers chemically and forms cross linking polymers where the mechanical properties become better. The incorporation of violacein pigment with membranes gives a slight effect on the physical structure and morphology of the membranes from both methods: casting and electrospinning. The tensile strength of electrospun membranes is higher, the difference in the spectra is highly observed, the surface-to-volume of the membrane is high because of fibre membrane, and the positive result in antibacterial activity test. The cast membrane is physically bonded where the spectra from FTIR do not gives huge differences. The release rate of casting increased with time and reached constant at the fifth hour while electrospun release rate decreased with time and remained constant at the third hour. The absorptivity of electrospun membrane is higher as compared to cast membranes In conclusion, the electrospun membrane has better physical and mechanical properties for wound dressing. The electrospun membrane is able to absorb high volume of pigment, high elasticity and has antibacterial activity detected on the membrane.

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