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Fabrication, characterization and release profile of aloe vera extracts/PVA composite electrospun nanofiber

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ABSTRACT

Aloe vera is well known remedies that carries many beneficial health effect such as painkiller, anti-inflammatory, promote skin growth and repair. Combination of bioactive natural product of aloe vera as a drug model and poly vinyl alcohol (PVA) as the base material or carrier in the electrospinning process were studied. Smooth straight and continues electrospun fibers were collected with the FESEM images show no formation of bead (defect signed) in the electrospun membrane when the concentration was set at 10% w/w of PVA nanofibre. Morphological structure of the electrospun membranes shows smooth and longitudinal fibre when aloe vera mixed with PVA polymer with percentage ratio of aloe vera over PVA less than 25%. FTIR shows no reaction between PVA and aloe vera by not showing peaks other than the initial materials. DSC proves the presence of aloin indicates the presence of aloe vera in nanofiber. The release profile of the electrospun aloe vera in PVA show higher initial burst release at 50% and 70% concentration levels which shows very little control of release or nothing at all. These results shows the potential of aloe vera – PVA electrospun nanofibers membrane as a promising material for wound dressing and topical drug delivery.

Keywords: Aloe Vera, Concentration, Drug release, Electrospinning and PVA

1. INTRODUCTION

Polyvinyl alcohol (PVA) known as non-toxic, biodegradable and synthetic polymer derived from polymerization of vinyl acetate followed with subsequent hydrolysis of acetate group to hydroxyl moleties [1]. PVA is a linear polymer having the formula $[CH_2CH(OH)]_n$ with a simple chemical structure of pendant hydroxyl group as shown in Figure 1. PVA also known as a "green polymer" due to its solubility and degradability. Furthermore, various natural materials can be easily combined with it and has a good compatibility with numerous polymer which further extend the range of its applicability [2]. As the properties of PVA is hydrophilic which easily dissolve in aqueous solution, it has also exhibit good chemical and thermal stability. Because of its excellent properties, PVA is widely applied in drug delivery system and dressing for clinicians where it has the ability to create and maintain moist wound environment as to improve the healing process [3]

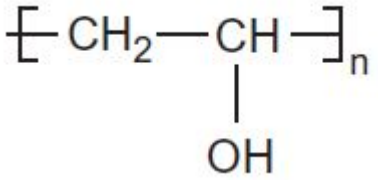

Figure 1: Chemical structure of Polyvinyl Alcohol (PVA)

PVA is a common choice in the preparation of various membranes and hydrogels [4]. Other studies were conducted where both PVA based cyrogel and hydrogel were successfully fabricated which PVA cryogel for transdermal drug delivery and PVA/sodium alginate hydrogel for wound healing applications [5,6]. Interesting studies by Sirima [7] when investigating the release of aloe vera extract in both electrospun PVA membrane and PVA hydrogel. They found that the swelling degree of PVA nanofiber membrane were higher than PVA hydrogel film which could be due to the fibrous structure of PVA electrospun fibres with higher surface area are capable of releasing more aloe vera compare to hydrogel film. Saja etal.[8] also reported a highly porous structure of the electrospun PVA membrane that disintegrate promptly which makes it an effective oral fast-dissolving drug delivery. All these findings were further strengthened the validity of electrospun nanofibers which were produced here via electrospinning.

Electrospinning is a well-known method to fabricate polymeric or melts nanofibers with the capability of producing fibers in the range of nano to micrometers . Straight forward and effective method that only require basic equipments including a DC high voltage power supply, a syringe pump together with the syringe and conductive metal needle and a collector connected with the counter electrode from the needle has gain much attention because of its ability to fabricate continuous polymeric ultrafine fibre or fibrous structures. In addition, electrospinning can be said as a method that can be further developed for mass production of continuous nanofibers for industrial applications [9]. The electrospinning process started with electrification of the droplet extruded from the spinneret (syringe). The droplet will soon be deformed into a conical shape (Taylor cone) which then when the electrostatic forces overcome the surface tension of the droplet, a liquid jet ejects and elongates in a straight line [10,11]. As the jet travels to collector, it exhibits into bending instability that stretching as far and elongate to form continuous electrospun nanofibers. The jet that travels would have solidified when it accumulates on the ground collector. In addition, following whipping and spiral pathway would reduce the fibre diameter, hence became thinner [12]. Electrospun nanofibers can be used as a biomimetic nanofiber with high specific surface area, high porosity, remarkable mechanical and biological properties, which makes them suitable for applications such as tissue engineering, drug delivery, and wound healing [13-15].

The usage of electrospun fibers in the field of biomedicine are mainly drug-controlled released, biological dressing and also tissue repair. In this project, aloe vera has been chosen to be the natural drug for the study of its delivery properties. Aloe vera or aloe *Barbadensis* is known as the oldest therapeutic herb that can be used to treat burn area on the skin. The health benefits of aloe vera plant have been spread throughout the world and records of the use of Aloe Vera as folk medicine date to antiquity with an early account from around 1500 B.C. As reported by Kumar et al [16] , aloe vera were used since the ancient civilization for the treatment of many diseases such as wounds treatment, reduce inflammation and mostly known can be used to treat injured tissue. Aloe vera as a bioactive natural product with a lot of medical properties which came from the active functional components of acemannan and glucomannan contained in the gel inside the leaves that can speed up wound healing through activating skin macrophages [17]. Further investigation also been done by Teplicki et al [18] suggests aloe vera can speed-up wound healing by promoting the proliferation and migration of fibroblast and keratinocytes that protecting from preservative-induced death.

Thus, this paper is discussing the aspects of encapsulation of Aloe Vera through the electrospining of polymeric nanofibre. Results are presented starting with morphological structures of electrospun nanofibers membrane. The chemical bonds features in the electrospun PVA-aloe vera composite nanofibers have been identified via FTIR analysis. Thermal analysis has

carried out to analyzed nanofibers crystallinity. On delivery properties UV-Vis Spectrometer was used to analyzed the samples.

2. EXPERIMENTAL METHOD

2.1 Chemicals dan raw materials

Polyvinyl Alcohol (PVA) at (Mw: 125 000g/mol) were received from Sigma-Aldrich and Aloe Vera Dry Extract was obtained from A&T Ingredient. Distilled water was used as the solvent to dissolve both the PVA and Aloe Vera. All chemicals were used as it is with no further processing and modification.

2.2 Samples Preparation

Part 1
Samples preparation were started with the polymeric PVA solution. Optimization of the PVA solution concentration as the carrier which will then be mixed with the aloe vera for electrospinning is crucial. Initially PVA powder was first weighted and dissolved into distilled water at 6% w/w and the mixture was stirred while heating up at 80°C. The prepared PVA (6%w/w) solution will then mix with aloe vera extract powder starting with 5% w/w content ratio for the start.
Part 2
The concentration of PVA in distilled water was then changed to 10% w/w and were mixed with varied aloe vera concentrations as tabulated in table 1. Each time, the solutions were carefully mixed and stirred until it become homogenous and crystal-clear solution before the electrospinning process.

Table 1: Solution mixture PVA-Aloe Vera in (% percentage)

No	Aloe Vera concentration (%)
1	0
2	5
3	25
4	50
5	75

2.3 Electrospinning Process

Electrospinning setup (figure 2) consist of a DC high-voltage power supply (Gamma BP series), syringe pump for non-medical which can provide feeding capacity 0-60 ml/h, a syringe with a blunt tipped needle connected to the positive electrode. A rotating drum acted as collector was covered with aluminum foil and connected to the negative electrode. A varied concentration of

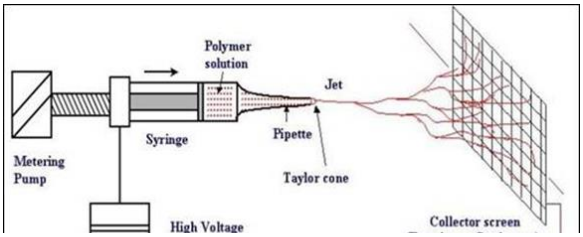

Figure 2: Schematic diagram of the electrospinning setup

AV in PVA were tested as tabulated in table 1. The electrospinning process will continue until fair amount of thick nanofiber mat (membrane) was able to be collected which normally took between 6 to 8 hours. The drum winder collector was fixed at a rotational speed of 50 rpm. Samples were oven dried for more than 12 hours for later stage of characterization and testing.

2.4 Samples characterization

Morphological structure of the electrospun PVA and PVA-Aloe Vera nanofibre were scanned using the Field Effect Scanning Electron Microscope (FESEM) by Zeiss with working voltage of 2 kV. All samples undergone gold-coating evaporator treatment to avoid any charging effect for 3 minutes before the imaging FESEM analyses. The average diameter was measured via Image J software.

2.5 Release of Aloe Vera

The electrospun membranes can be separated-off from the aluminum foil that covered the drum winder collector and cut into certain sizes then weighted on electronic balance. Roughly about 0.03 g of each sample were measured. For the start, each sample were immersed in 40 ml of distilled water with magnetic stirrer at 300 rpm in ambient temperature of 37 ^{0}C. When the membrane had been dissolved completely in the solution, a small amount of it was taken out to determine the presence of aloe vera using UV-Vis spectrometer at an optical length of 299 nm. About 5 ml was withdrawn at a given time intervals and subsequently replaced with similar amount of fresh water in order to maintain the constant sink condition. These procedures were repeated for each of four samples.

3. RESULTS AND DISCUSSION

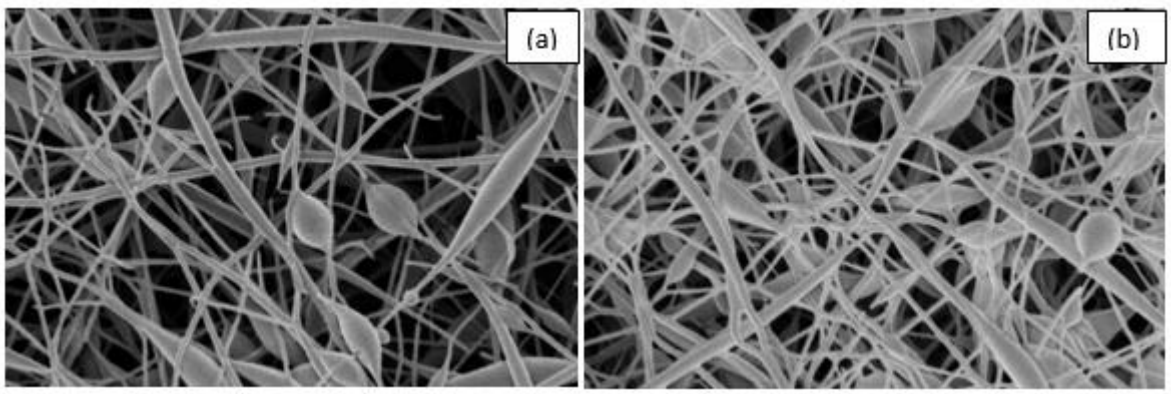

Figure 2: (a) As spun 6% PVA (b) Composite 6% PVA – 5% Aloe Vera

As discuss earlier finding optimum conditions for electrospinning such as solutions mixture of PVA – water to be mixed with aloe vera were carried out. Figure 2 shows the image of SEM of: (a) as-spun nanofiber at 6% of PVA-water (b) the 6% PVA-water solution was mixed with 5% w/w Aloe Vera. In this preliminary experimental works, important variables that can affect the resulted morphology of the electrospun fibers such as the applied voltage, nozzle-collector distance and feed rate were set here as; 12.2 kV, 20 cm and 0.13 ml/h respectively. The resulted electrospun morphological structures for both solutions show formation of beads which was a sign of weaknesses and not a favorable structure for biomedical applications. However, the average size (diameter) of beads was decreasing from 542nm to 430nm when Aloe Vera extract

powder were added. The fibre density also increased which indicates that the presence of Aloe Vera is somehow has improved the electrospun fibers yielding. The viscosity and conductivity of the solution were thought to be the reason behind the overall improvement. Higher concentration of aloe vera will results in the increasing of the ionization capacity of the aloe vera-PVA solution which also responsible in lifting the electrical conductivity. It is a well-known fact that high electrical conductivity in the solution will ease up the drawing of jet stream in the electric field.

To test further on the precursor solution, the concentration of PVA solution mixed with aloe vera powder was slightly increased from 6% w/w to 10% w/w. After testing the range of possible variables, the optimum conditions are set as followed: voltage at 15kV, feed rate = 0.5 ml/h and distance needle – collector = 15 cm. Figure 3 shows the SEM micrographs of the electrospun aloe vera-PVA composite nanofibers. Smooth and bead-less electrospun nanofibers were successfully collected when the PVA-water solutions concentration was changed from 6% to 10%. Polymeric solution concentration is one of the most critical processing parameters where generally higher concentrations will produce larger fibers diameters.

Shape of fibers is also depending on the processing parameter where variety of shape such as round fibers, flat ribbons fibers, bead fibers etc. can be produced by an electrospinning process. Figure 3 also shows us that by changing the ratio of PVA-aloe vera several shapes starting from round shape and flat ribbons followed with beaded fibers. Branched fibers and split fibers were among shapes that can be spotted in almost all concentration ratio. Beads and pores are common features found in electrospun fibers. Beads are normally appeared at the higher solution viscosity (concentration), and in our case it starts to appear when the solution ratio between PVA-aloe vera is at (25%-75% w/w) level. Beaded fibers were also become dominant once the ratio of the aloe vera surpass the 25% mark. Solution viscosity which in our case proportional with content of aloe vera were varied to find the optimum concentration which results smooth and straight fibers. It was suggested the balance between electrostatic forces and surface tension of the sol that normally produces smooth and straight fibers is disturbed which resulted beaded fibers.

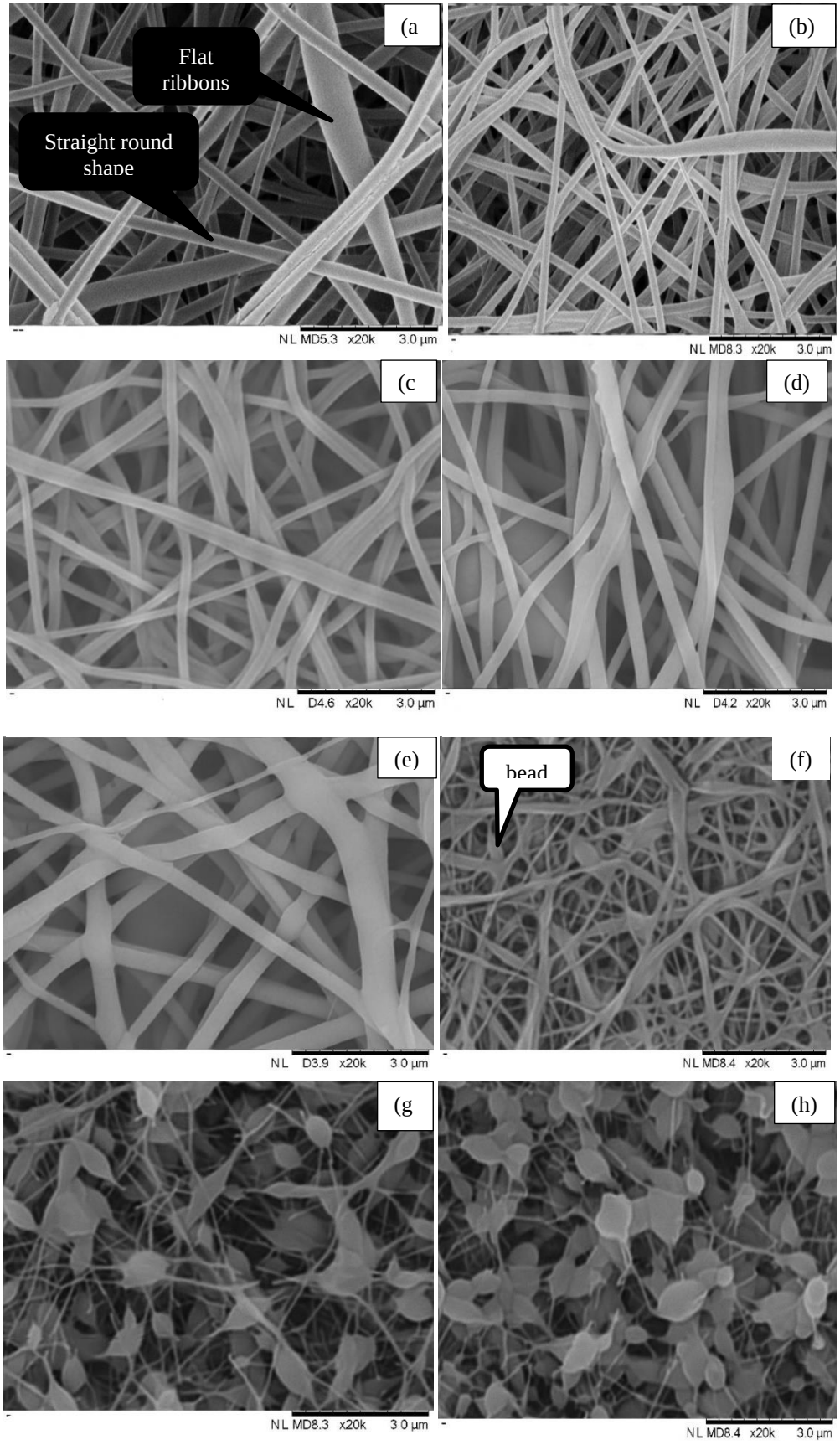

Figure 3: SEM micrograph of the electrospun aloe vera (AV)-PVA composite nanofibers; a) AV 0% w/w, b) AV 5% w/w, c) AV 10 %w/w, d) AV 15 %w/w, e) AV 20% w/w, f) AV 25% w/w, g) AV 50 % w/w and h) AV 75% w/w .

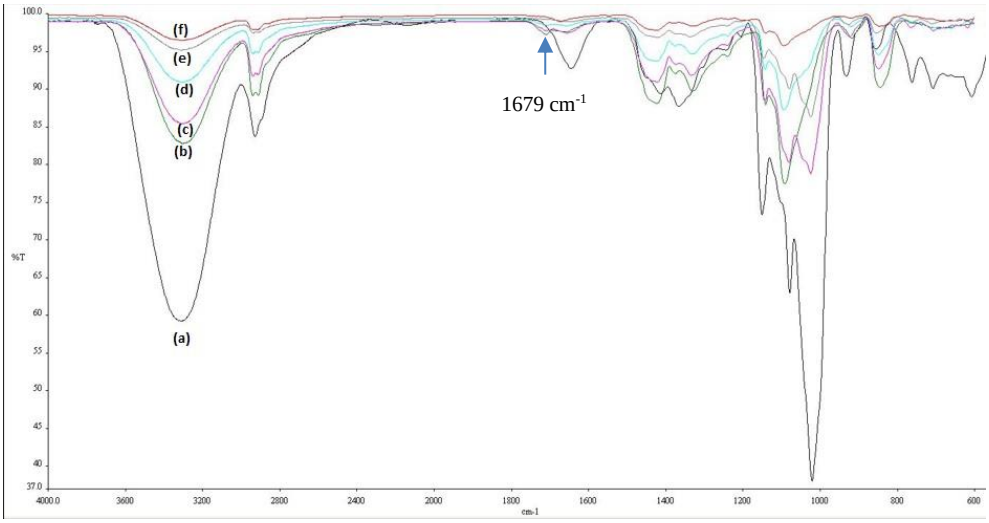

Figure 4: FTIR spectrum for (a) aloe vera extract powder; aloe vera's content in the electrospun fibers (b) AV 0% w/w, (c) AV 5% w/w, (d) AV 25% w/w, (e) AV 50%w/w, (f) AV 75% w/w.

The FTIR spectra in figure 4 shows the aloe vera (powder) and PVA-aloe vera composite electrospun fibers chemical composition. Strong and broad intensity band appeared at 3480 cm^{1} in aloe vera powder indicates phenolic-Oh group in aloe vera [19]. The peak also appeared at 3400 cm-^{1} – 3380 cm-^{1} in both PVA and PVA-aloe vera where it shows the majority characteristic of their respective assignment which represent hydroxyl group in the membrane. It is due to the properties of PVA and aloe vera that both contain hydroxyl group. Such appearance also been recorded by Li et al [8], where there is a presence of water absorbed (-CH_3) stretching by PVA molecular chain. Apart from that, Peresin et al [20] and Zaman et al [21] also recorded major peak that related to hydroxyl group and acetate group which responding to neat PVA. The peak at 2780 cm-^{1} appear due to the stretching of hydroxyl group which present in both polymer-drug. This situation also been studied by Azabayjian et al [22] where hydrogen abstraction occurred in between the mixture of PVA and T_4 which slightly change the characteristic of polymer due to intermolecular interaction between the hydrogen group with carbonyl in both PVA chains and aloe vera. These can be seen when the peak at fig. 4 (c) shift from 1679 cm-^{1} to 1690 cm-^{1}. According to Akhagari et al [23] and Awanthi et al [24] there was no any significant change while combining the polymer and drug either in ionic and hydrogenic even irradiation to promote the presence of the drug which assumes the good prominent method of network formation. But the distinguished of peak in the PVA-AV does not affect the efficiency as drug carrier as most of the characteristic were remained unchanged.

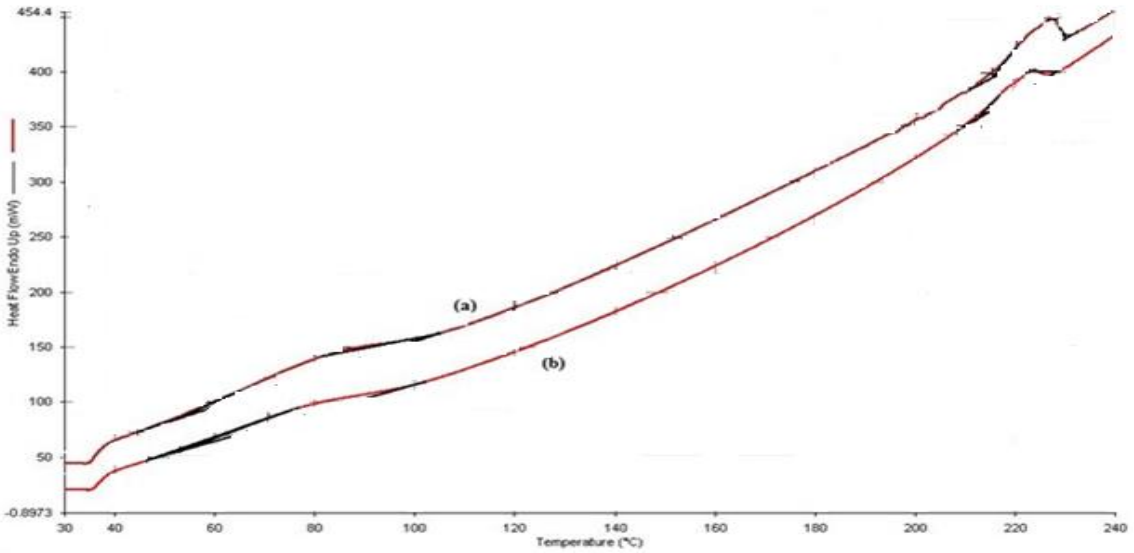

Figure 5: DSC thermograms for nanofibre of (a)electrospun PVA 100% and (b) electrospun PVA- aloe Vera

Figure 5 shows differential scanning calorimetry (DSC) thermogram of electrospun the PVA 100% fibers and PVA-aloe vera fibers. The electrospun PVA fibers exhibit the loss of moisture ranging at 60˚C to 100˚C as it shows the peak at 81˚C of glass transition. It started to melt when reaching 222˚C then possibly thermal degrades between 240˚C to 300˚C. The alignment and semi-crystallizations of polymer chain in individual PVA fibres had demonstrate the high sheer stress during electrospinning. For comparison, when aloe vera was mixed with PVA polymer, the electrospun fibers began to loss their moistures earlier at 76˚C. This was possibly because of the presence of aloin in aloe vera[25]. The presence of aloe vera might affect the crystallinity of neat PVA polymer which convert into amorphous structure that easier to degrade as reported by Khanzada. H [33]. Combination of aloe vera and PVA shows a good miscibility which also signaling the lacking of crystallinity[26]. The melting point was also decrease from 226˚C to 222˚C which is another strong evident of amorphous structure. Consequently, the increase of macromolecules mobility subject to polymeric chain due to the present of aloe vera has previously demonstrated [27,28] where single endothermic response was found probably due to the amorphous state. The result of thermogram had elucidated as well that aloe vera-PVA electrospun fiber was successfully fabricated which can be a promising material for wound dressing and topical drug delivery.

Release percentage of the aloe vera from PVA electrospun nanofibers matrixes were tested using the UV-Vis spectrometer (Figure 6). At 60s, the percentage of Aloe Vera reached around 30% to 40% and increased steadily until 70% at 150s which suggests the immediate release of drug had occurred during degradation from PVA membrane [29]. The fastest release of aloe vera from the PVA electrospun nanofiber matrix was the 75% aloe vera content where after just 240s almost the entire content were released. At 75%, the electrospun aloe vera- PVA membrane had not offered any signs of controlled where burst released was occurred soon after it was exposed to water. At 50% some interesting results were found where some control over the release of aloe vera where flat curves can be seen at around 80s and 200s respectively and this trend of controlled were maintain for the other remaining ratios. The immediate release of Aloe Vera probably because of the hydrophilic properties which is very soluble in the water since the experiment was using water as the medium transport. Moreover, high surface area and

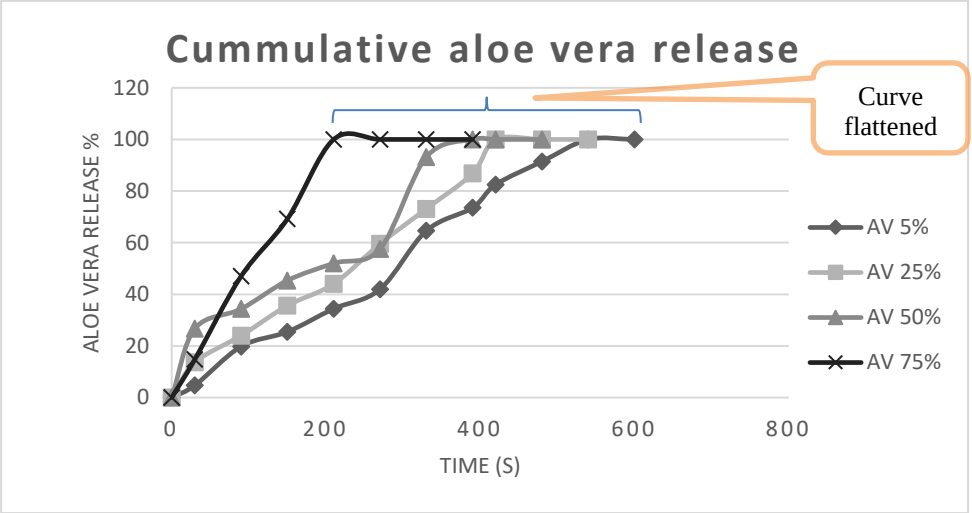

Figure 6: Percentages delivery rate of aloe vera from the PVA electrospun nanofibers nanofibers matrix

porosity obtained through small diameter of as spun nanofibre also promote fast release of the drug. Not only that, the excellent wettability in nanoporous structure remarked the rapid penetration of water, thus shorten the release time [30]. Taepaiboon et al [31] stated that the highly porous structure of as-spun nanofibre had contributed to immediate release of drug whereas according to Lee et al [32], releasing rate of PDGF-BB from heparin conjugated polycaprolactone (PCL) /gelatin scaffolds was propotional with fiber diameter whereby smaller diameter fibers showed faster release. Initially, the immediate release of Aloe Vera provides a quick onset of action at specific site but according to R.Imani et al [37], sharp increase of drug concentration potentially increase the toxic levels of drug at initial drug administration which probably cause high therapeutic effect that might slowdown the healing process. However, at 5% of Aloe Vera content, the drug was steadily release on time taken which suggested that it was release on controlled manner thus reduced the toxic level on the surface of targeted cell. In addition, formation of beads at 50% and 75% of Aloe Vera content also give an initial burst release as both of composite membrane show the fastest release in figure 6. The drug release behaviour of beads had also been studies extensively by T.Li et al [36] where it concluded that electrospun bead-on-string could potentially be used to attenuate the drug initial burst release. Nonetheless, both of Aloe Vera content at 50% and 75% were not suitable candidates because of high percentage of drug release on short period of time that might cause high therapeutic effect on specific target area on cell.

4. CONCLUSION

This paper aims to fabricate the electropun PVA and PVA-Aloe Vera nanofibre as polymer-drug carrier were successfully achieved. The morphological structure of electrospun PVA nanofibre shows smoother and non beaded nanofibers when the concentration were increased from 6% to 10%. Both FTIR and DSC displayed the presence of Aloe Vera as an evident to prove the encapsulation of drug in polymer membrane. Aloe vera release profile have shown very encouraging results where smooth and non-beaded fibers demonstrate an ability for controlled release of aloe vera from PVA matrixes. Electrospun fibers of Aloe vera-PVA with concentration 50% and 75% have shown burst released with no flat curve detected especially for 75% sample which indicated only a little control of release or nothing at all. Both 50% and 75% samples morphologically are beaded fibers with some small fraction of fibers which suggest the importance of fine and smooth electrospun nanofibers to limit the burst release that can cause an

increase in toxic level on the target surface which also causing therapeutic effect that might slowdown the healing process.

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