A Protocol for Measuring Pull-off Stress of Wound-Treatment Polymers

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Skin wounds and burns compromise the body's natural barrier to bacteria and other pathogens. While many forms of wound dressings are available, polymeric films are advantageous for various reasons, ranging from the ease of application to durability. One common drawback of using polymeric films for a wound bandage is that the films tend to adhere to common inanimate objects. Patients spend hours in contact with soft and hard materials pressed against their skin, which, if the skin was dressed with a polymeric film, would inflict further wound damage upon body movement. In this work, we present a novel technique that allowed for measuring polymeric tackiness, after a long incubation period, with materials regularly encountered in a hospital or home setting, and soft fabrics. The polymers were exposed to an environment intended to simulate daily conditions and the technique is designed to perform multiple experiments simultaneously with ease. Four commercially available polymers (new-skin, no-sting skin-prep, skin shield, and Silesse) were tested as proof-of-concept to gather preliminary data for an overall assessment of wound treatment efficacy, resulting in the estimation of pull-off stress of the polymers from a specimen of porcine skin. Silesse did not reveal a measurable tackiness, no-sting skin-prep had the highest mean tackiness (13.8 kPa), while the mean tackiness between new-skin and skin shield was approximately equal (9.8 kPa vs. 10.1 kPa, respectively), $p = 0.05$. Future work on polymeric fluids for wound dressing applications should include tensile stress and dynamic viscosity estimations. [DOI: 10.1115/1.4027412]

Keywords: wound treatment, bioadhesion, polymeric films, mechanical testing, tackiness

1 Introduction

The skin is the largest organ in the body and is responsible for protecting the internal organs from infection [1]. Skin injury can range from superficial damage to the epidermis, to complete degradation of the dermis and subcutaneous tissue as a result of

severe scrapes and burns. Burn injuries are a common form of postcombat skin damage experienced by soldiers [2] and are particularly susceptible to infection [3]. Wound treatment strategies must provide sufficient water and oxygen permeability, and protect against bacterial invasion [1]. Synthetic polymers are common for soft tissue repair application and are available in many forms: polyurethanes, Teflon®, Proplast®, methyl methacrylate, silicon, etc. Fast curing polymers can be applied quickly in a hostile environment and provide a protective barrier in the form of a sterile homogeneous polymeric network structure that can vary in thickness. Commercial polymeric films are also commonly utilized in civilian hospitals and are even available for household use.

A common problem with polymeric dressings intended for wound treatment is that the cured films tend to stick to objects (e.g., chairs, toilet seats, bed sheets) that are common in households and hospital settings. To this end, when comparing among the commercially available polymeric films that are applied to the epidermis, it is important to consider the modes of failure of polymer detachment and the stress imposed onto the skin. Ideally, after curing, a given film should adhere to the skin and present minimal tackiness to inanimate objects (metals, plastics, threaded sheets). Tackiness, a technical term for “stickiness” [4], is intuitively understood as the sensation felt when struggling to remove ones finger from a substance and is the result of adhesion. Adhesion is defined as a bond between an adhesive and a solid substrate, which requires more than the typical surface energy ($0.01\text{--}0.1\text{ J/m}^2$) to separate. An industrial adhesive would require between 100 and 1000 J/m^2 of energy for separation [5]. Tackiness is, therefore, defined as the resistance that must be overcome to separate two solids that are joined by a liquid (or cured) adhesive [6]. In the case of a cured adhesive, the resistance to separation is identical to the adhesive's mechanical strength, assuming that it is greatly exceeded by the adhesion between the substrates. For this reason, tackiness is mostly a function of the adhesive's rheological properties and is independent of the molecular bond formed by the adhesive and a solid substrate [6,7]. Nevertheless, when considering a wound treatment function, the mechanisms of adhesion are complex. For example, the polymer is applied onto the skin in a liquid state and has the opportunity to occupy the rough texture of the epidermis, thus resulting in a high adhesive bond strength. Theoretically, the polymer is cured when making contact with an external inanimate surface and thereby achieves a significantly lower state of adhesion. For this reason, failure is likely to occur as an instability in the polymer's structural integrity or a separation at the interface of the polymer and the inanimate object.

There are three general modes of detachment: pull-off, shear, and peel [4,5]. Most biomedical research investigating tackiness of wound treatment modalities are focused on peel with the objective of investigating polymer bioadhesive strength [8,9]. Nevertheless, pull-off detachment normal to the skin surface is a likely scenario in actual patient-specific applications, which has been largely overlooked by the medical community. A technique to investigate the bioadhesive strength of patches, developed by Wong et al. [10], was applied on the surface of a chicken pouch using a texture analyzer [11,12]. For several other skin treatment adhesives, tack has been evaluated using pull-off probe tests [13–16] and ball-rolling tack tests [8,17–19]. Tack assessment is also often useful in other applications [17] such as food processing, evident by the work of Chen et al. [20] who utilized a tensile probe separation method to measure the adhesion force between different foods and a 40 mm diameter aluminum probe.

In the present work, we describe an apparatus and outline a protocol for measuring the normal pull-off stress of polymeric films intended for wound treatment. In addition, four commercial polymeric solutions are subjected to the protocol to demonstrate proof-of-concept and compare treatment efficacy. Moreover, we demonstrate how simple modifications of the proposed apparatus can be used to measure adhesive properties between polymeric

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Fig. 1 Pull-off testing apparatus and nomenclature: (a) pull-off stress testing assembly configured for the Bose Electroforce T3200 testing apparatus; (b) radial insert ball bearing system; and (c) close-up of the Block-Skin-Polymer-Dolly assembly clamped to the load cell top plate and metal stoppers.

films and any commonly encountered material, such as soft fabrics. Noteworthy is that the objective of this work is not to measure the bioadhesive strength of any substance, but rather to quantify the tackiness (or normal pull-off stress) of a wound dressing polymer that has been cured between skin and a material that a patient is likely to be in contact with in an everyday setting (e.g., steel, plastic, and soft fabrics). Furthermore, the novelty of the proposed technique is that it allows to re-create a realistic environment that can vary depending on the intended application of the polymeric film.

2 Methods

A uniaxial mechanical testing device (Electroforce T3200, Bose Corp., Minneapolis, MN) was utilized as a general support structure, a means for high accuracy and high volume data acquisition, and a constant-rate electric displacement actuator. Figure 1 shows a comprehensive assembly of the proposed pull-off testing apparatus with a nomenclature of the subcomponents. The overall assessment of tackiness occurs between the cured outer surface of a polymeric film adhered to porcine skin and aluminum dolly (shown in Fig. 2(a)). The objective is to allow the dolly's flat surface to adhere to the polymer under preset conditions, and measure the peak force upon detachment. Furthermore, we demonstrate how the technique can be expanded to measure tackiness of the polymer for most soft fabrics. Four commercially available polymers in liquid form were tested as a proof-of-concept of the proposed protocol: New-Skin (Prestige Brands Holdings, Inc., Tarrytown, NY), No-Sting Skin-Prep (Smith & Nephew, Inc., Andover, MA), Skin Shield (Insight Pharmaceuticals Corp., Langhorne, PA), and Silesse (ConvaTec, Inc., Skillman, NJ).

2.1 Pretest Sample Preparation Protocol. A preprocessing step subjects each tested bond to a simulated environment likely to be encountered in a biomedical application. Multiple samples are incubated under identical environmental and mechanical force conditions, thereby minimizing measurement variability that is likely to arise from sample pretreatment inconsistencies. Moreover, the method is particularly efficient by offering the opportunity to perform multiple tests simultaneously.

Porcine hide samples were harvested from a local butcher and stored at a temperature below 4 °C for 24 h. The hide was then cut into 6.5 × 6.5 cm squares and placed on wooden blocks wrapped

Fig. 2 (a) Schematic showing primary testing components by layer. The load cell top and bottom plates are made of Plexiglas® and machined to accommodate the load cell threading. (b) A picture of weight distributed over 12 samples curing inside an incubator.

in foil to prevent swelling. The epidermal surface of each square was lightly colored with food dye to visualize detachment at the polymer-dolly interface during the experiment. 300 ml of the polymer was applied in liquid form using a laboratory grade pipette and allowed to dry for 30 min. Upon application, each

Fig. 3 Protocol for conducting pull-off testing between polymeric films intended as wound dressings and soft fabrics: (a) a clamp is laid atop a flat piece of pig hide with polymer applied; (b) a dolly and cloth are compressed onto the skin through the clamp. This assembly can be made simultaneously and placed in incubation as shown in Fig. 2; (c) a custom made part containing a threaded shaft; and (d) as the assembly is placed on the bottom base for testing, the custom part (shown in c) is pressed through the clamp and the fabric is secured to the shaft.

polymer tested in the proof-of-concept experiments would generally coat the skin surface with a relatively even thickness. The skin-polymer-dolly assembly (see Fig. 2(a)) was then placed in an incubator for 3 h at 37 °C, with each dolly compressed on the hide at approximately 2.0 kPa (Fig. 2(b))—equivalent to the stress exerted by an average male on a bed in a supine position [21]. The wooden blocks inside the incubator were laid on a porous Styrofoam® cushion and submerged in a column of 5 mm of water, which allowed the water vapor to engulf the testing samples and provide an even weight distribution across all dollies.

2.2 Pull-Off Test Using Soft Fabrics. Figures 3(a)–3(c) show a modification to the protocol for measuring tackiness between polymeric films and soft fabrics. It is often necessary to quantify tackiness with soft fabrics as clothes are likely to be in direct contact with the dressing and patients are sometimes confined to a hospital bed for long periods of time. After applying the polymer, a Jubilee Hose Clamp was used for each hide sample (see Fig. 3(a)) to house the soft fabric and a dolly under compression (see Fig. 3(b)), which can then be incubated as in Fig. 2(b). As the sample is placed on the testing apparatus, the dolly is removed and the stage is raised, fitting the conduit of the clamp around the 20 mm diameter shaft (Fig. 3(c)), until a sufficient compressive force is achieved. Finally, the clamp is raised and firmly tightened around the groove machined onto the shaft to secure the fabric (Fig. 3(d)).

2.3 Pull-Off Test by Dolly Retraction. The Block-Skin-Polymer-Dolly assembly was taken out of the incubator one sample at a time and clamped to the top of the load cell top plate (Fig. 1(a)—Clamp 1) with metal stoppers (Fig. 1(c)). The mean time elapsed from removing the assembly from the incubator to retracting the Dolly was less than 90 s. The manual stage was positioned to align the Dolly with the radial insert ball bearing system and clamped to the base plate (Fig. 1(a)—Clamp 2). The Dolly was guided by adjusting the elevation of the manual stage into the radial insert ball bearing system until the force transducer displayed a noticeable compression. As the head of the Dolly head was verified to be well within the bearing system, the locking collar was engaged. Finally, the Dolly was retracted to a maximum separation distance of 1 cm, at a rate of 10 cm/s, resulting in 0.1 s for the duration of the pull-off test.

2.4 Data Analysis. The load cell used for the present experiments was of a 22 N capacity (Honeywell 34T, Honeywell,

Columbus, OH). Load cell tension data were collected at 20 Hz and postprocessed in MATLAB (Mathworks, Inc., Natick, MA). The force of detachment was considered to be the maximum force measured during the experiment, while the work of separation was calculated as the integral under the force vs. displacement curve. One-way analysis of variance (ANOVA) with a significance level of $p = 0.05$ was used to confirm the following null hypothesis: the mean tackiness and work of separation are equal among the four different commercially available polymeric films tested.

3 Results

Sample pull-off tests were conducted for four commercially available polymer wound dressing films: new-skin, no-sting skin-prep (S&N), skin shield, and Silesse. Figure 4 shows the failure modes of these polymers, which are largely consistent between experiments of the same product. For simplicity, we define three general modes of detachment: (1) failure in the adhesive strength at the polymer-skin interface; (2) failure in the adhesive strength at the polymer-dolly interface; and (3) failure in the structural integrity of the cured polymer. Interestingly, all four polymers revealed a consistently different mechanism of detachment. New-Skin shows the second and third mode of detachment, while skin shield mostly resulted in the first and third modes. S&N appears to be characterized by all three, while Silesse only failed according to the second mode.

Figure 5 illustrates the mean and standard error of the maximum pull-off stress imposed on the skin before detachment for New-Skin, S&N, and Skin Shield. The insert in Fig. 5 shows an example of the raw data collected from the uniaxial testing apparatus. Silesse did not reveal a measurable tackiness and is thus not shown in this figure. S&N revealed the highest mean tackiness to aluminum (13.8 kPa), while the mean tackiness between new-skin and skin shield was approximately equal (9.8 kPa vs. 10.1 kPa, respectively). Excluding the data on Silesse, all pull-off stresses were processed through the ANOVA statistical algorithm, which rejected the null hypothesis with $p = 0.05$.

The mean and standard error of the work required to separate the Dolly from the Block-Skin-Polymer-Dolly assembly are shown in Fig. 6. Consistent with previous findings, S&N requires the most work (37.8 J/m²), while the other two polymers are relatively equal (24.9 J/m² for New-Skin and 28.6 J/m² for Skin Shield). Excluding Silesse from the statistical analysis, we found no statistically significant differences in the work of separation of these three films ($p = 0.12$).

4 Discussion

Polymeric films are commonly employed as a wound dressing strategy to prevent infection and facilitate healing. A common concern amongst patients and health care professionals is that most commercially available films have a propensity for adhering to common household items. We present a protocol for measuring polymer tackiness that is capable of simultaneous testing of multiple samples and robust to consider any readily available material (including soft fabrics). To our knowledge, there has been limited work focused on the pull-off stress measurements of polymeric wound treatment options. For biomedical applications, the novel advantage of the described apparatus is that the probe is detached from the moving actuator. Therefore, multiple samples can be pretreated in a simulated environment prior to the pull-off stress assessment. In experiments where each testing sample must be cured and pretreated in contact with the detaching probe for long periods of time, the radial insert ball bearing system with a locking collar overcomes the difficulty of attaching the probe to an actuator without compromising the cured adhesion. The current protocol can be implemented by simple reconfiguration of a uniaxial mechanical testing apparatus and the accuracy improved by using a load cell sized for the expected pull-off loads. The four

Fig. 4 Appearance of dolly and skin after pull-off experiment for the 4 polymeric films. All 3 modes of detachment are present among the films: (1) failure in the adhesive strength at the polymer-skin interface; (2) failure in the adhesive strength at the polymer-dolly interface; and (3) failure in the structural integrity of the cured polymer.

Fig. 5 Mean and standard error of pull-off stress with skin for three commercial products: New-skin (9.8 ± 0.8 kPa), no-sting skin-prep (13.8 ± 1.4 kPa), and skin shield (10.1 ± 1.4 kPa). The insert shows exemplary Force vs. displacement data for skin shield. Silesse is not shown as it revealed no measurable tackiness.

commercially available polymeric films were tested for tackiness when in contact with aluminum.

We compared the typical mode of separation failure among the 4 wound dressing films. The second mode of failure (i.e., failure in the adhesive strength at the polymer-dolly interface) appears ideal for biomedical applications. The dressing remains intact and fully adhered to the skin. Nevertheless, even in this mode, exposing the wound to stress as a result of slight adhesive bonding between the polymer and a metal surface could still be uncomfortable and painful for the patient. After curing, which is almost instantaneous, Silesse revealed no measurable pull-off stress making it an ideal wound dressing product as it relates to pull-off discomfort. This is likely due to the fact that Silesse appears to be a polymeric fluid, while the other three products are polymer film-formers. Silesse is applied to the skin as a pressurized mist; S&N is applied as a spray-on, and New-Skin and Skin Shield are brushed on. The four dressing options revealed statistically significant ($p=0.05$) differences in pull-off stress. Considering that an ideal film should have adhesiveness to the skin and not to inanimate objects, Silesse has these ideal characteristics. However, it

Fig. 6 Mean and standard error of the work of separation with skin for three commercial products: New-skin (24.9 ± 2.4 J/m²), no-sting skin-prep (37.8 ± 5.4 J/m²), and skin shield (28.6 ± 4.6 J/m²).

resulted in the thinnest dressing among the four products and should be subject to mechanical testing against known properties of elasticity for human skin.

The compliance of the skin substrate lowers the force required to achieve adhesive separation because only certain regions are in tension at a given time [5]. In fact, if the gap between the metal stoppers and dolly surface was increased, the recorded pull-off stress should theoretically decrease. For this reason, when testing soft fabrics, the compliance of the fabric will undoubtedly be a critical property in the mechanics of separation. In addition, the retraction rate of the dolly in the experiments likely influences the ensuing pull-off stress and should be analyzed in future studies as part of a parametric analysis approach.

We presented tackiness data for polymers that were incubated at 37°C for 3 h. Nevertheless, patients that are confined to a hospital bed could place the wound dressing under constant compression, and in contact with bedding or other surrounding materials, for well over 8–12 h. The proposed method allows for further testing to determine how the duration of incubation affects tackiness. To assess each film's efficacy as a wound dressing barrier, full mechanical characterization testing must be completed. In future work, we will apply the current technique in combination with

mechanical and rheological testing to determine the benefits and drawbacks of commercially available polymers for wound treatment. Design characteristics considering patient comfort and treatment reliability should be analyzed across relevant characteristics ranging from delivery methods to shape retention.

5 Conclusion

A novel protocol was described for measuring tackiness between polymeric films intended for wound treatment and practically any material commonly encountered in a hospital or household setting, including soft fabrics. Before testing, each sample undergoes compression under environmental conditions designed to simulate a real life scenario. As proof-of-concept, we measured tackiness in the form of pull-off stress and work of separation for four polymeric films that are currently available for wound treatment applications. Silesse reveals no measurable tackiness, while the mean tackiness for the other three films (new-skin, no-sting skin-prep, and skin shield) was statistically the same for the work of separation, unlike the pull-off stress which revealed statistical differences among the products. In future work, the protocol will be expanded to account for assessment of the tensile and viscous properties of the polymers.

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