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Standardized methodology for in vitro assessment of bone-to-bone adhesion strength

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ABSTRACT

Advancement of adhesives technology has been limited in orthopedics, which still has a striking reliance on metal hardware to help facilitate fracture healing. Despite an obvious clinical need, bone adhesives are not currently available on the market. Testing the bone adhesion strength and other aspects of the adhesive performance is extremely complex. This paper presents standardized methodology for in vitro assessment of bone-to-bone adhesion strength. Two test configurations (lap shear and butt joint) were used to comparatively assess the adhesion strength of four commercially available adhesive material, poly(methyl methacrylate) cement (CMW), glass-ionomer cement (FUJI), dimethacrylate resin (SB) and cyanoacrylate resin (VB), which were allowed to set under two environmental conditions (air and water). Under dry conditions, both test configurations generally yielded similar measurements of adhesion strength, which was around 1.1, 2.8 and 9.1 MPa for CMW, FUJI and VB, respectively. The dry adhesion strength for SB measured using the butt configuration (2.5 MPa) was 43.2% higher compared to that measured using lap shear (1.4 MPa). In a wet environment, the measured adhesion strength generally decreased and was dependent on the test configuration used. The failure mode of the samples adhered using CMW was adhesive, while that using FUJI, SB and VB was a combination of both adhesive and cohesive, independent of the test configuration and the setting condition. This proposed methodology is comparable to ASTM standards and can be used to study the effect of different biomaterial formulations as well as test parameters on the bone-to-bone adhesion strength, in a reproducible manner.

1. Introduction

Bioresorbable bone adhesives have the potential to mitigate concerns related to metallic fixation devices, which often necessitate invasive removal surgeries, increasing patient burden and draining valuable hospital resources [4–6]. Despite an obvious clinical need, bone adhesives are not currently available on the market. There are significant challenges for bonding living tissue, such as bone, mainly due to the wet environment, aggressive immune system, and an exhaustive list of requirements including the biocompatibility of the adhesive and its degradation products [4,6]. Most importantly, bone adhesives must form a bond strong enough to be useful and sustainable long enough, typically three months, to allow fracture healing. Although it is extremely difficult to quantify the required level of adhesion, which strongly depends on the application and anatomical location, Weber and Chapman [7] suggest a practical lower limit of 0.2 MPa, below which reduction is difficult to maintain.

Testing the bone adhesion strength and other aspects of the adhesive performance is extremely complex. The former is typically measured in vitro and/or ex vivo using animal tissue, whereas in vivo studies tend to focus on the functional aspects of the procedure and the biological response associated with the adhesive. There is a lack of consensus on how to measure adhesion to bone and methods reported in the literature [8–12] as well as values of adhesive bond strength have varied widely [13,14]. This is mainly because the tests used not only measure the effect of stress and environment on the adhesive, but also measure other parameters associated with the test itself. Furthermore, it is extremely difficult to mount bone tissue directly into the grips of the testing machine and achieve reproducible test geometries due to the inherent variability associated with the irregularities of the bone [15]. This paper presents a robust and repeatable methodology for in vitro assessment of bone-to-bone adhesion strength. The aim is to compare the relative effectiveness of different adhesive material as bone adhesives and help study the effect of various test parameters and environmental conditions on bone adhesion.

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2. Materials and methods

2.1. Sample preparation

Femoral bovine bone was obtained from a local butcher and cut using a fine tooth wood band saw into approximately 100 × 15 × 10 mm (L x W x D) rectangular sections, which were then sawn in half to create two 50 × 15 × 10 mm complementary bone samples. Subsequently, the samples were rinsed under running water to remove any loose soft tissue and stored in the freezer until testing.

2.2. Experimental protocol

A hole was drilled into one end of each bone sample along the anterior-posterior direction and the surface of the opposite end was smoothened using first 120 then 800 grit abrasive paper to achieve comparable surface roughness and ensure that the surfaces of the bone samples to be adhered match and lie flat against each other. In this study, the adhesion strength of four commercially available material, CMW (CMW1 Radiopaque, DePuy Synthes, PA, USA) poly(methyl methacrylate) bone cement, FUJI (Fuji IX GP, GC America Inc., IL, USA) glass-ionomer cement, SB (Scotchbond™ Universal, 3 M Company, MN, USA) dimethacrylate resin, and VB (Vetbond™, 3 M Company, MN, USA) cyanoacrylate resin, was estimated using two test geometries, lap shear (Fig. 1) and butt joint (Fig. 2).

The constituents of the tested adhesive material are presented in Table 1. All adhesive material were mixed as per the manufacturer's recommendations (refer to Table 2) and applied onto the smoothened bone area to achieve the configurations shown in Figs. 1 and 2. Plastic clamps were used once the biomaterial was applied onto the bone surface to fasten the bone samples, achieve comparable biomaterial thickness and aid in bond formation. Excess biomaterial surrounding the adhered area was removed using a spatula. Two different setting conditions were tested to study the effect on the adhesion strength: 1) the biomaterial was allowed to set in air at room temperature (17.3 ± 0.5 °C and 50.9 ± 1.9% humidity) then tested immediately after and 2) the biomaterial was allowed to set in water at 37 °C overnight then tested immediately after. Once the biomaterial set in the desired environmental condition, the adhered bone sample was fixed into one of the aluminum fixtures using potting material (Bondo®, 3 M Company, MN, USA). Visual inspection was used to ensure vertical and concentric alignment in the potted sample. The fixture (with the potted sample) was then placed into the top grip of the material testing machine (Instron 3344, Instron Corporation, MA, USA) and slowly lowered into the bottom fixture containing freshly mixed potting material. The potting material was allowed to harden for 20 min before the sample was loaded in tension at a crosshead control rate of 1.0 mm/min until failure.

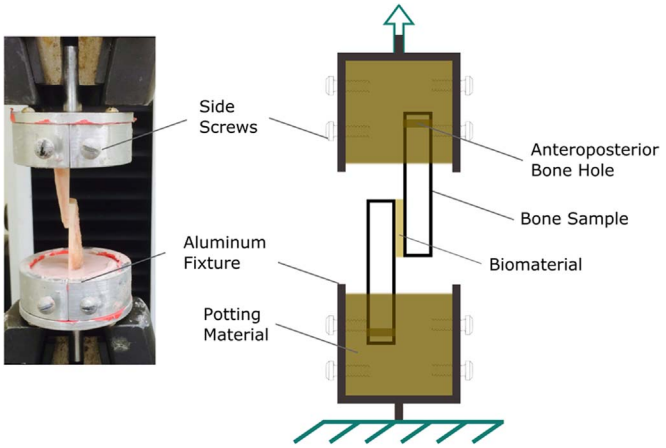

Fig. 1. The experimental set-up for measuring the biomaterial adhesion strength using the lap shear configuration. (Left) Photograph showing the lateral view of the actual bone sample in the top and bottom grips of the testing machine. (Right) Schematic showing the lateral view of the adhered bone sample fixed in the top and bottom fixtures.

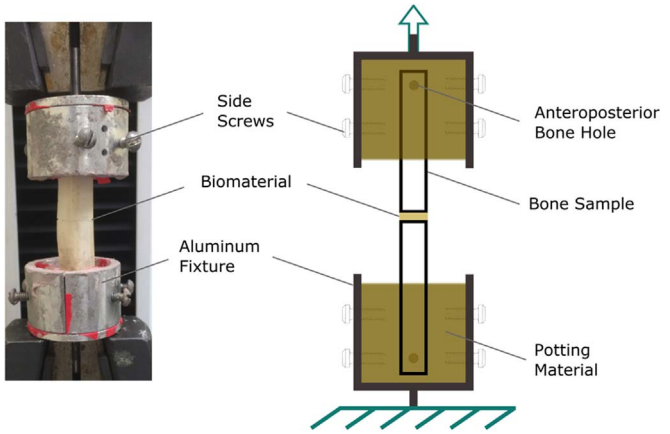

Fig. 2. The experimental set-up for measuring the biomaterial adhesion strength using the butt joint configuration. (Left) Photograph showing the frontal view of the actual bone sample in the top and bottom grips of the testing machine. (Right) Schematic showing the frontal view of the adhered bone sample fixed in the top and bottom fixtures.

Table 1
Constituents of the adhesive material used in this study.

Biomaterial	Composition
CMW 1 Radiopaque (CMW)	Poly(methyl methacrylate), benzoyl peroxide, barium sulfate, methyl methacrylate, N,N-dimethyl-p-toluidine, and hydroquinone
Fuji IX (FUJI)	Polyacrylic acid, aluminum fluorosilicate glass, tartaric acid and water
Scotchbond Universal Adhesive (SB)	Dimethacrylate resins, methacryloxydecyl phosphate monomer, HEMA, Vitrebond™ copolymer, ethanol, silane, and water
Vetbond Tissue Adhesive (VB)	N-butyl cyanoacrylate, hydroquinone and blue dye

In all experiments, the peak force at failure was recorded. The bone samples were digitized and the photos were processed in imageJ [16] to measure the adhesion area. The adhesion strength was then calculated by dividing the peak force at failure by the adhesion area. All experiments were repeated six times and the data were presented as mean ± standard deviation. The influence of the biomaterial formulation, the test geometry and the biomaterial setting environment on the adhesion strength was evaluated using the independent-samples t-test with a significance level $\alpha = 0.05$. All statistical analyses were performed in SPSS version 23 (SPSS Inc., IL, USA). Furthermore, in order to determine the failure mode, the surface morphology of representative samples was assessed using a scanning electron microscope (SEM, MIRA3 LM, TESCAN, Brno, Czech Republic) with an acceleration voltage of 20 kV. Energy-dispersive x-ray spectroscopy (X-Max 80 mm^2, Oxford Instruments, Oxfordshire, United Kingdom) was also used to confirm the presence or absence of the biomaterial on the bone surface.

2.3. Validation study

The aim of this validation study was to compare the methodology presented in this paper to ASTM D1002. The lap shear test was repeated using polyurethane bars (Sawbones, WA, USA) with a rectangular geometry of 100 × 30 × 3 mm (L x W x D). The geometry of the polyurethane bars allows the adhered samples to be placed directly into the grips of the testing machine according to ASTM D1002 standards (Fig. 3). This testing method will be referred to as “No-Fix” since no aluminum fixtures were used to mount the samples into the