Novel Electro-spray Technique for Applying Phospholipid Coatings to Titanium

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Statement of Purpose: Phospholipid coatings on titanium implants have been shown to enhance osteoblast activity, promote mineralization, and facilitate implant osseointegration in vivo.^{1,2} The advantages of phospholipid coatings compared to other biomimetic techniques include significant enhancement of osteoblastic mineralization and the associated reduction of inflammatory response. Dip and drip coating techniques for applying phospholipid coatings have been used on titanium to date. Both coating techniques are easy to perform, but can present difficulties in controlling the amount of material on the final surface, resulting in coatings too non-conformal and thick for in vivo use.²

Electro-spraying (e-spray) is a method of atomization of a liquid by means of electrical forces. The technique involves spraying a source material at a controlled rate onto a target material under the influence of a high voltage differential between source and target. Electro-spraying provides the advantage of being able to create coatings with relatively high efficiencies because the electrical charge difference "carries" the liquid source material, which also provides good control of coating morphology, especially on rough and intricately shaped surfaces.³ Other advantages of this technique are low cost, easy setup, and the range of materials that can be used as coatings. The purpose of this study was to develop an e-spray technique for applying phospholipid coatings on titanium implants.

Methods: The e-spray technique was developed using the phospholipids DSPC (1,2-Distearoyl-*sn*-Glycero-3-phosphocholine) and DOPS (1,2-dioleoyl-*sn*-glycero-3-phospho-L-serine). Both were chosen based on prior osseointegration studies, with DOPS clearly being the best for osseointegration. However, DSPC is considerably less expensive so it was used in initial technique development. The phospholipids were both dissolved in chloroform, to a concentration of 5% w/vol for DSPC and a concentration of 2.5% w/vol for DOPS.

A syringe (10 cc, glass, Hamilton) containing the phospholipid/chloroform solution was mounted on a syringe pump. A double hub syringe tube (12 inch, 20 gauge, Hamilton) connected the syringe to the needle (22 gauge, blunt end). Target titanium samples (flat, commercially pure Ti, 0.016 thickness, 1cm x 1cm) were mounted with silicone putty on an insulated circuit board with copper wires protruding from the back through the board, directly contacting the Ti samples. This target board was positioned at a measured distance from the needle. The needle and the copper wire backing the target board (contacting the Ti samples) were connected to the positive and negative (ground) poles, respectively, of a controlled voltage source. The pump, syringe tube, syringe body and mounting hardware were also grounded.

As the syringe pump was activated, source material sprayed from the syringe needle as a very fine, almost indiscernible mist onto the target material.

Results: The best parameters for the e-spray process were found to be in the following ranges: pump rate - 6 ml/hour; distance - from 8 to 12 cm; voltage - 8 to 12 kV. It was also found that the most consistent (between Ti samples) surfaces were created by spraying between 3 and 5 Ti samples at once, oriented radially on the target board.

Figure 1 shows SEM images of DSPC coatings on Ti samples. Coatings are consistent across the Ti sample. Rod shaped structures are clearly visible, and their packing density varies with spray voltage.

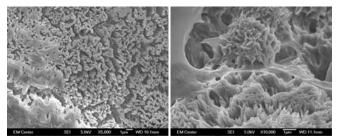


Fig. 1: *SEM images of DSPC coating on Ti 5 keV, at 5,000X (left), and* 10,000X (right) original magnification. Scale bars are 1 μm.

Figure 2 shows SEM images of DOPS coatings on Ti samples. Coatings are conformal and consistent across the Ti. Surface morphologies were modified as shown (left to right) by reducing the spray voltage 2000 volts and the spray distance by 2 cm.

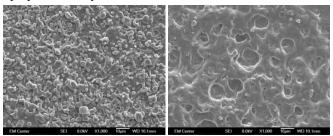


Fig. 2: SEM images of DOPS coating on Ti 8 keV, at 1,000X (left), and 1,000X (right) original magnification. Scale bars are 10 μm.

DSPC surfaces washed off the Ti samples in less than 4 hours in phosphate buffered saline (PBS). DOPS surfaces remained on some Ti samples after 2 days of PBS rinse. **Conclusion:** This work demonstrates that electro-spraying is an effective technique for creating thin, consistent and conforming phospholipid coatings on Ti surfaces. We have demonstrated that varying spray parameters will produce different surface morphologies that may prove useful in controlling *in vivo* response – i.e., cellular response may be manipulated by surface roughness). DOPS surfaces are more durable than DSPC surfaces, probably due to the inherent greater polarity of the DOPS molecule relative to the DSPC molecule.

References:

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- 3. Chen, C.H. et al. Thin Solid Films, 1999. 342(1-2): p. 35-41.