MICROENCAPSULATION OF OCTYLCYANOACRYLATE AS THE HEALING AGENT FOR SELF-HEALING BONE CEMENT

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ABSTRACT

Previous research has confirmed that the elegant encapsulated healing agent approach developed by White et al. [1] can be utilized to restore the material strength and function of composite systems. However, existing designs do not employ materials that adhere to the constraints presented by biomaterials, a field that could greatly benefit from the application of this method of self-healing. A variety of implants, including those designed for orthopaedic, cardiovascular, and dental applications, fail following the accumulation of microdamage sustained over lifetimes of cyclic loading.

Cemented joint replacements are subject to failure via microcrack formation that results in the production of wear particles and subsequent loosening of the implant; these particles become caught in the joint space, increasing abrasion and accelerating the rate of wear [2]. The addition of an encapsulated healing agent to the poly(methyl methacrylate) (PMMA) bone cement could toughen the matrix and provide the means to repair microcracks, thereby minimizing the production of wear particles and extending the lifetime of the implant. Medical grade cyanoacrylates used in sutureless surgeries react with water and anions present in the body and are viable candidates for the encapsulated healing agent; no additional catalyst would need to be incorporated into the matrix, simplifying the material design.

For this project, interfacial polymerization of polyurethane (PUR) with a 1,4-butandiol chain extender was used to encapsulate octylcyanoacrylate (OCA). Pluronic F68 (2% by weight) was dissolved in deionized water while PUR and OCA were dissolved in separate volumes of methyl ethyl ketone (MEK) and combined into a single organic phase just prior to addition to the stirring aqueous phase. 1,4- butanediol was added dropwise to the emulsion and was allowed to react with the PUR for 1h at 40°C. The protocol was repeated as the agitation rate of the emulsion was varied from 250 to 1100rpm; the average diameter and standard deviation of the capsules were found to decrease with increasing agitation rate (Table 1). The average shell thickness ranged from 9.88 $\pm$ 4.88µm to 3.24 $\pm$ 1.42µm, with thicker shells observed at lower agitation rates.

Table 1: Average capsule size and standard deviation at various agitation rates.

Agitation Rate (rpm)	Average Capsule Diameter (µm)	Standard Deviation (µm)
250	513.05	290.10
500	219.68	73.88
700	166.44	46.51
900	110.84	29.26
1100	89.30	20.94

A basic test of the reactivity of the encapsulated OCA was performed by crushing the capsules between two glass slides to visualize release of the capsule contents and to verify these contents glued the surfaces together. Thermogravimetric analysis (TGA) was also used to determine the content of the capsules 1 day post-fabrication (Figure 1) [3]. TGA results indicated that the capsules were composed of 51-59% OCA and 34-40% PUR by weight. Trace amounts of solvent (up to 7%) were also present in the final capsules. TGA experiments performed following 1 month dry storage of capsules produced at 700rpm indicated a reduction in core material of 4.4%; experiments will be repeated following 3 and 6 months dry storage to analyze the changes in the composition of the capsules over time.

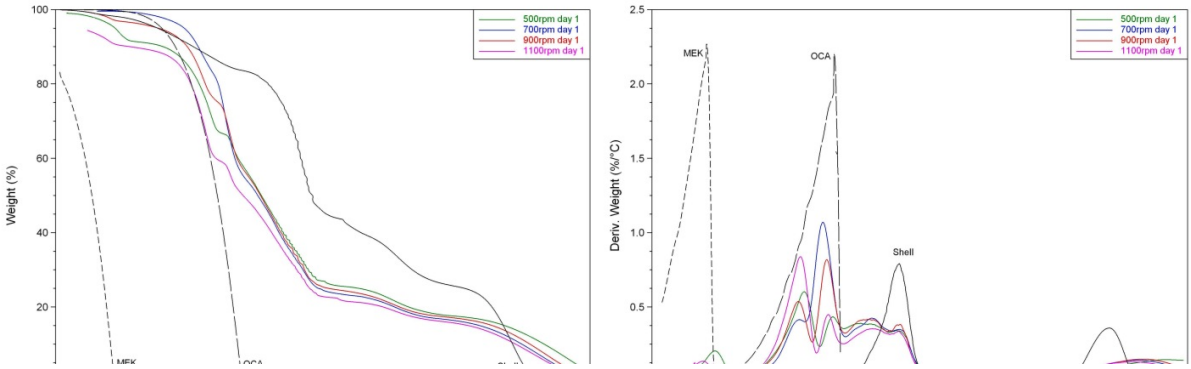

Figure 1: (A) TGA weight loss curves of capsules and (B) their derivatives.

TGA of residual fluid from the encapsulation protocol performed at 500rpm indicated its decomposition curve closely resembled that of deionized water. Approximately 87% of the OCA sample still remains when heated to 170°C (Figure 1A), however, 98% of the residual fluid sample had already decomposed before reaching this temperature, suggesting the amount of OCA present in the residual fluid is minimal.

Based on the analysis of the residual fluid, a larger amount of OCA will be used in the encapsulation protocol to increase the efficiency of OCA encapsulation and possibly the volume present in the resulting capsules. Future work will focus on the mechanical testing of individual capsules [4] followed by the incorporation of these capsules into a PMMA bone cement matrix and subsequent testing of the matrix.

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