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Modification of two Universal Adhesives by Incorporating Ascorbic Acid Coated Superparamagnetic Fe_3O_4 Nanoparticles

(Comparative *In Vitro* Studies)

A Thesis Submitted to the Council of the College of Dentistry, University of Baghdad, in Partial Fulfillment of the Requirements for the degree of Doctor of Philosophy in Restorative and Aesthetic Dentistry

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Abstract

A major concern associated with contemporary dental adhesives is their insufficient capability to completely infiltrate and penetrate into the deepest parts of the demineralized dentin collagen matrix, thereby; over-time, they would be susceptible to hydrolytic and enzymatic degradation affecting their dentin bond strength and bonding durability. Thus, the aims of this study are to prepare, characterize, and incorporate a colloidal suspension of Ascorbic Acid coated Superparamagnetic Nanoparticles (AA-SPN) into two universal adhesives (All Bond Universal, Prime&Bond Universal) to enhance their penetration deeper into the dentin substrate by subjecting them to external magnetic force to increase their dentin bond strength and durability and to investigate their effect on the cytocompatibility, degree of conversion, water sorption/solubility, and antimicrobial activity of the adhesives.

In this study, a colloidal suspension of AA-SPN was prepared and characterized by atomic force microscopy, transmission electron microscopy, and Fourier transform infrared spectroscopy that clearly showed the uniformity of particles size distribution in the range of 18.7±2.4 nm, and the nanoparticles were entirely coated by the ascorbic acid. The AA-SPN was then incorporated into the adhesives at different mass percentages. The 2% AA-SPN was selected as the best percentage to be incorporated to the adhesives according to the results of the preliminary studies. Then several experimental studies were conducted in which four adhesive groups were tested, i.e., Group I: All Bond Universal (control), Group II: 2% AA-SPN incorporated All Bond Universal, Group III: Prime&Bond Universal (control), and Group IV: 2% AA-SPN incorporated Prime&Bond Universal.

The 2% AA-SPN incorporated adhesive groups showed significantly higher bond strength values (both immediate and after thermocycling / 6 months water aging) in response to the applied magnetic force than the controls. Group IV showed significantly higher values than Group II, whereas Group I showed the lowest bond values followed by group III. Those results totally agree with the results of the scanning electron microscopy study regarding the resin tags penetration depth and hybrid layer thickness.

The cytocompatibility of the 2% AA-SPN incorporated adhesives was tested by the MTT assay and the multi-parametric HCS assay on fibroblast-like cell line and

showed a slightly higher cells viability than the control groups with no significant differences among the groups.

The degree of conversion (DC) of the 2% AA-SPN incorporated adhesive groups in comparison to control groups was tested both immediately and 24-hours post-curing and showed no significant differences. Group IV showed significantly higher DC than Group II, while Group I showed the lowest DC followed by group III.

The water sorption study showed no significant differences among the 2% AA-SPN incorporated and the control groups. However, Groups III and IV showed significantly higher water sorption values than Groups I and II. While regarding the solubility, the 2% AA-SPN incorporated groups have significantly lower values than the control groups. Group IV showed significantly lower solubility than Group II.

The microbiology study showed that the 2% AA-SPN incorporated groups have significantly higher inhibition zones against (*Streptococcus mutans* & *Lactobacillus acidophilus)* than the control groups. Group IV showed significantly higher inhibition zones than Group II, and there was no significant difference among the control groups.

In conclusion, incorporation of AA-SPN into the two universal adhesives enhanced their infiltration deeper into the dentin substrate in response to the applied magnetic force resulting in a significant enhancement in their bond strength and durability. Also, it enhanced their antibacterial activity and reduced their water solubility without negatively affecting their cytocompatibility or degree of conversion.