Republic of Iraq
Ministry of Higher Education
and Scientific Research
University of Baghdad/ College of Dentistry
Department of Conservative and Esthetic
Dentistry



## Assessment of Two Universal Dental Adhesives Modified by Fluorinated Graphene Nanoparticles (A Comparative in Vitro Study)

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 Conservative Dentistry

By

Rafal Mohammed Mariush B.D.S., M.Sc. (Conservative Dentistry)

Supervised by

**Assist. Professor** 

Dr. Abdulla M.W. Al-Shamma

B.D.S., M.Sc., Ph.D. (Conservative Dentistry)

2020 A.D. 1442 A.H.

## **Abstract**

**Background:** to investigate the enhancement of the adhesive-dentin hybrid layer by the addition of fluorinated graphene nanoparticles (FGN) into two types of commercially available universal adhesive, guided using the total etching technique as a bonding procedure.

**Experimental adhesives preparation:** Addition 2% of silanated FGN into two brands of universal adhesives: AllBond Universal adhesive (Bisco, USA) and Prime&Bond universal (Dentsply, USA( by using a magnetic string for 30 min followed by ultrasonication for 1h.

Materials and Method: Specimens were allocated into four groups. Group I (control group): using the non-incorporated AllBond Universal adhesive. Group II (experimental group): using 2% of FGN incorporated AllBond Universal adhesive .Group III (control group): using the non-incorporated Prime&Bond universal adhesive. Group IV (experimental group): using 2% of FGN incorporated Prime&Bond universal adhesive.

**Cytotoxicity test:** was done by mosmann's tetrazolium toxicity assay and multiparametric cytotoxicity assay direct contact to fibroblast cell line. The results showed that all adhesive systems with 2% of FGN were biocompatible and there was no systemic toxicity.

**Fourier Transform Infrared Spectroscopy:** was used to ensure the presence of fluorinated graphene nanoparticles (FGN) by testing the FGN before and after mixing with AllBond Universal and Prime&Bond universal adhesives. The results showed that the silanted FGN was interacted with the adhesive materials.

**Shear bond strength test:** was done on flat occlusal sound dentin which was obtained by cutting through the occlusal enamel and was done by utilizing laryee

universal testing machine. The results were showed significantly the highest values with group II and group IV than the control groups.

**Fluoride ion release test:** was done by immersion of 45 discs of experimental adhesives (group II and group IV) into dioinized water for 1, 3, 7, 14, 21, 28, 35, 42 and 60 days and the release of fluoride ion was measured into the immersion solution in ppm using ion selective electrode. The results showed higher release of fluoride at the first and third day and then was decreasing subsequently through the tested periods until be stable at the 42 and 60 days with statistically non-significant difference between the two experimental adhesives groups (p>0.05).

**Microbiology study:** was carried out against *Streptococcus mutans* and *Lactobacillus acidophillus* by using agar well diffusion technique which was involved ten wells made in petrri dish filled with modified adhesives and then measuring the inhibition zone by using a digital ruler. The results showed that all experimental adhesives had antibacterial effects against *streptococcus mutans* and *Lactobacillus acidophillius* with a significant difference between control and experimental groups.

Microleakage test: was done by linear dye penetration test on the buccal and lingual surfaces, preparation of a standardized class V cavities were prepared in the buccal and lingual surfaces of (3mm height, 3mm width, 2mm depth)of lower third molar in which the cervical margins of the cavities were located about 1 mm occlusal to the cementoenamel junction. All the specimens were subjected to thermocycling procedure (500 cycles), and then stored in container containing 2% Methylene Blue for 24 hours at room temperature. The two segmented halves of specimens were measured the linear dye penetration by using stereomicroscope under a magnification of 45x. After analyzing data, the result showed that the linear dye penetration whether occlusally or gingivally were significant and lowest

leakage was shown by group II followed by group IV with no significant difference among experimental groups.

Scanning Electron Microscope& EDX test: was used to compare the hybrid layer thickness and the resin tags lengths inside the dentinal tubules for the control and the experimental groups after mixing with 2% of FGN and elemental contents evaluation chemically by used EDX. Sixteen third molar teeth were used for this experiment then sectioned in buccolingual direction. The restoration – tooth interface was studied and photographed by SEM at (1000X, 3000X, 5000X) magnifications. The results of resin tag lengths and hybrid layer thickness were show statistically non-significant difference between the control and experimental adhesives groups (p>0.05).

In conclusion, incorporation of fluorinated graphene nanoparticles into the two universal adhesives enhanced their mechanical properties resulting in a significant enhancement in their bond strength and durability. Also, it enhanced their antibacterial activity and fluoride ion release without negatively affecting their cytocompatibility or wettability.