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SHEAR BOND STRENGTH OF NUSMILE- NEOPUTTY BIO- CERAMIC MATERIAL TO FOUR DIFFERENT RESTORATIVE MATERIALS USED IN PEDODONTICS

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ABSTRACT

Objective: The study aimed to assess the shear bond strength of four restorative materials that are widely used in pediatrics to NuSmile- Neoputty material. The restorative materials were two glass ionomers: Fuji II LC and Equia Forte Fil, and two dental resin composites: Cention N and Venus Bulk Fill.

Materials and methods: A total of 164 specimens were used in the study. 160 specimens were used for the shear bond strength testing and failure mode analysis. Four specimens were needed for scanning electron microscopic investigation of NuSmile surface with and without application of MDP containing adhesive. Specimens were divided into four groups according to the restorative material bonded to NuSmile: Group I: Fuji II LC. Group II: Equia Forte Fil. Group III: Cention N. Group IV: Venus Bulk Fill. Further subdivision into two subgroups (A & B) was done according to application of the adhesive or not respectively.

Results: Subgroups (IA) and (IIIA) revealed significantly higher shear bond strength values than other groups with no significant difference between them, followed by subgroup (IVA). Subgroup (IIA) revealed the least bond strength value.

Conclusions: Fuji II LC glass ionomer and Cention N composite would be capable for inducing stronger bond with NuSmile- Neoputty compared to Venus Bulk Fill composite. On the contrary, the bond strength of Equia Forte Fil to NuSmile- Neoputty could not be considered satisfactory. Application of MDP containing adhesive can enhance the bond strength to Nusmile- Neoputty regarding Fuji II LC, Cention N, and Venus Bulk Fill.

Keywords: NuSmile- Neoputty, Fuji II LC, Equia Forte, Cention N, Venus Bulk Fill.

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INTRODUCTION

For the preceding several years there was a large variety of bio-ceramic materials applied in vital pulp therapy dental procedures of deciduous teeth, taking the mineral trioxide aggregate (MTA) as a starting base. The satisfactory mechanical properties of MTA, and its superior chemical characteristics, as well as the high clinical rate of success had supported its wide use. On the other hand, its high staining potential was considered a challenging drawback that should be overcome. Also, its bonding to tooth structure and to different restorative materials was considered an important aspect seeking improvement.[1]

Manufacturers had presented different MTA products. Among these products is NeoMTA plus, which was proven to have a success rate comparable to MTA in pulpotomy of deciduous teeth. NeoMTA plus was further modified to NeoMTA2, which is a stain-free MTA. The resistance to staining was achieved by using tantalum oxide as a radiopacifier instead of bismuth oxide "which is the cause of discoloration".[2]

NuSmile- Neoputty MTA was introduced by Nusmile Inc. Houston, TX; USA, that was approved by the U.S. Food and Drug Administration (FDA) in 2020. It is a premixed, resin free bioactive bio-ceramic MTA that can trigger the formation of hydroxyapatite, and thus can promote dentin bridge formation. It had used the same tri and di- calcium silicate based powder as in NeoMTA2. A previously conducted study in 2022 by Ozata et al.[3] revealed that the shear bond strength of Neoputty and NeoMTA2 to bulk-fill dental resin composite was similar, supporting the capability of its wide field of application, giving high preference in pediatrics for most of pulp treatment modalities.

NuSmile- Neoputty was described by the manufacturer to have many preferable characteristics; as being of high radiopacity, non toxic, and of high resistance to staining. It has putty- like and non-tacky consistency, in addition to its satisfactory immediate wash-out resistance. The immediate wash- out resistance of Neoputty permits the opportunity of performing the restoration immediately after its placing. Also, gives the ability of immediate cementation of a crown. Neoputty has different indications than being a root canal sealer; thus neither high flow nor low film thickness is required. Neoputty delivers a ready-to-use material for direct placement with no need for mixing, and formulated to set in the oral environment by the action of intra- oral moisture. Multiple uses are offered by NuSmile- Neoputty material; as dental procedures contacting vital pulp tissues in both direct and indirect pulp capping. Also, it is indicated in pulpotomy, apexogenesis, as well as being used as a base under restorations. For a pulpotomy, base or pulp capping, a layer at least 1.5mm thick should be applied. In addition, it can be applied in dental procedures contacting peri-radicular tissues; as root perforation repair, apexification, and root-end filling procedure. For root apexification, the Neoputty has to be compacted in the apical region to create a 3 to 5 mm thick apical barrier. NuSmile- Neoputty is not recommended in cases of pulpectomy of deciduous teeth; since if a primary tooth requires pulpectomy and the permanent successor is absent, the primary root canals can be restored with gutta-percha and root canal sealer in an attempt to retain the primary tooth for long term.[4]

It was recommended by the manufacturer to apply a layer of a flowable composite, resin modified glass ionomer, zinc oxide and eugenol, or any other restorative material on top of the Neoputty, prior to application of the final tooth restoration; noticing that it is not mandatory. If a flowable composite will be applied and an etching step is required, then etching the tooth not the Neoputty material has to be performed.[4]

Regarding pediatric dentistry, there have been several varieties of dental materials for restoring

primary teeth. Some of which were proven to be successful, however others still require further assessment. In this line, investigating the use of NuSmile- Neoputty for either deciduous or permanent teeth in children and teenagers is of great importance. Additionally, it is a major issue to assess the new-generations of glass ionomer, as well as dental resin composite restorative materials that have a route of use in pediatric dentistry.

Adequate bond strength between bio- ceramic materials and restorative materials is considered a highly decisive factor for the success of vital pulp therapy. The shear bond strength of MTA based materials to some types of resin composites and glass ionomer restoratives has been evaluated in different studies using variable etching and bonding procedures. The previously conducted studies had reported a wide and conflicting range of shear bond strength values. Additionally, the shear bond strength of premixed bio- ceramics NuSmile-Neoputty to the nowadays used pediatric restorative materials has been considered in very limited studies. The dental pediatric literature, in particular, is still indistinct about the best restorative material to be placed on top of NuSmile-Neoputty MTA.[5, 6]

Glass ionomer has been always considered as a "gold standard" for restoring pediatric teeth. This is attributed to its unique properties; such as the release of anti- cariogenic fluoride and the ability of chemical bonding to tooth structure. On the contrary, glass ionomer showed some limitations due to its high susceptibility to fracture and the low wear resistance. These deficiencies had made it unsuitable for being applied in high stress bearing areas.[7]

Resin modified light cured Fuji II LC and packable glass ionomer Fuji IX are considered among the most widely used glass ionomer restorative materials. A previous study was conducted in 2019 by El Sayed at al.[8] to compare Fuji II and Fuji IX in relevance to marginal adaptation, anatomical form and marginal staining. The study concluded that Fuji II and Fuji IX showed comparable marginal adaptation. While, Fuji II restorations showed better results in relevance to preserving the anatomical form, incidence of secondary caries and occurrence of marginal discoloration when compared to Fuji IX, and consequently recommended the use of Fuji II LC as a restorative material in primary teeth. GC Fuji II LC capsule is a light-cured glass ionomer restorative with high flexural strength and excellent bond strength to teeth, even in presence of saliva. It is available as pre-weighed unit dose capsules, offering no need for mixing and prevents the loss of material. GC Fuji II LC with its acceptable esthetics, self adhesion properties, good moisture tolerance, and simple using technique had made it a preferable choice for class V restorations, cervical erosions, abfraction lesions and also as a base under restorations.[8]

The achievement of work with high accuracy and fast performance is an imperative value for a pediatric dentist. This can be attained by having materials that are easy in manipulation and of suitable working time. Recently, a new production of high-viscous glass ionomer cement (HV- GIC) has been introduced. First, Equia was launched in 2007 by GC Corp, and numerous clinical studies have demonstrated its high clinical efficacy in restoring class I and class II cavities.

In 2014, the new glass hybrid technology made more considerable improvements, leading to the development of Equia Forte. This restorative system had combined a self-cure, bulk fill restorative "Equia Forte Fil" laminated with a nano-filled, self adhesive resin coat "Equia Forte coat". This recent innovation represents the glass hybrid technology that was based on existence of glass fillers having different sizes.[9]

Equia Forte Fil contains ultra-fine, highly reactive, surface treated fluoro- alumino- silicate glass particles that are dispersed within the conven-

tional glass ionomer structure. It also contains a high-molecular weight polyacrylic acid. This material had shown an enhanced flexural strength, with high translucency and good esthetic properties. It had also offered improved flow characteristics with non-sticky handling properties. The nano-filled self-adhesive resin "Equia Forte coat" can induce optimized marginal seal and better wear resistance.[10]

Considering, the insufficient data regarding the clinical performance of Equia Forte on primary dentition, a previous study was conducted to compare and evaluate Equia Forte with Tetric N Ceram composite resin, in class II restorations in primary molars. The study concluded that restoring the stress bearing areas in primary molars using Equia Forte and Tetric N Ceram showed no significant difference regarding the clinical performance for a period of 12 months. Additionally, Equia Forte can be considered an acceptable restorative material in small to medium sized class II cavities in primary molars, especially in relevance to the ease of application in pediatric patients.[7]

Dental resin composite restorative materials had gained a wide range of practice in pediatric field of dentistry. The recently developed "alkaline" restorative material Cention N is considered a new form of tooth-colored bulk fill restorative material. It is dual cured; having urethane dimetacrylate (UDMA) and a self curing initiator. The liquid in Cention N consists of di- methacrylates and initiators. The powder consists of a mixture of glass fillers, initiators, and pigments. It is a radio- opaque material that contains alkaline glass fillers capable of releasing fluoride, calcium, and hydroxide ions to offer anti- cariogenity. Due to the presence of cross-linking methacrylate monomers, in combination with the effective self-curing initiator, Cention N has a high polymer network density and high degree of polymerization over the complete depth of the restoration. It can be utilized with or without a pre-adhesive step.[11, 12]

Another restorative composite that gained high attention was Venus Bulk Fill. It is a flowable, low-shrinkage posterior bulk-fill restorative dental resin composite. The material is ideal to be utilized in deciduous dentition. Venus Bulk Fill can permit easy filling of class I and class II cavities up to 4 mm increments. Regarding primary teeth, just one single increment of Venus composite filling material would be satisfactory. The reason behind the ability of 4mm depth of cure of Venus composite is its high translucency that permits strong light penetration. An additional special feature possessed by this composite material is its low viscosity, which allows self-leveling and self-adaptation to the cavity walls.[13]

The application of an adhesive for bonding of MTA based materials to dental resin composites gained a great confirmation in the dental literature. The 10- Methacryloyloxydecyl dihydrogen phosphate (MDP) containing universal adhesives were found to be able for creating a strong chemical bond with hydroxyapatite, by inducing surface dissolution of hydroxyapatite crystals; with subsequently formation of water resistant MDP- Calcium salts.[14] 3M Single bond universal adhesive is widely used in the dental practice. It contains Vitrebond copolymer (polyalkenoic acid: which is the copolymer used in light cured resin modified glass ionomer to provide a consistent bonding under either dry or moist conditions). Poly (alkenoic) acid is chemically known to be a family of complex acids that includes poly(acrylic), poly (itaconic), and poly(maleic) acid. In the presence of water, the COOH groups in polyalkenoic acid can possibly undergo partial ionization into carboxylate anions COO^- and hydrated protons. The negative carboxylate ions can react with calcium ions, inducing a strong chemical bond. 3M Single Bond Universal adhesive is hydrophilic before light polymerization. This hydrophilic character permits the adhesive to undergo proper wetting of the tooth surface, and so can penetrate into the dentinal

tubules. On the other hand, it is hydrophobic after being polymerized. This hydrophobic character can improve the hybrid layer integrity. Single Bond Universal adhesive contains an ethanol/ water-based solvent system; which is less volatile than acetone, and so helps maintaining a consistent viscosity, as well as satisfactory handling properties during usage. In addition to the solvent system of Single Bond Universal adhesive, the rest of the constituting formulation was optimized to avoid phase separation during its application. 3M™ Vitrebond™ copolymer, hydroxy- ethyl-methacrylate (HEMA) and water present in the formulation allow for enhanced bonding to etched dentin, even if the dentin surface is accidentally over- dried; since water can rehydrate the collapsed collagen and allow for the formation of a proper hybrid layer.[14]

Single Bond Universal adhesive had made use of the MDP monomer, in addition to integration of silane into its structural composition. The MDP monomer has been known to be able for bonding to zirconia and alumina, as well as to metals. The silane component can permit the adhesive to bond to glass containing materials. These two main components had made the Single Bond Universal adhesive gained its wide use with these substrates without requiring the application of a separate ceramic or metal primer prior to its placement.

The literature constitutes several studies concerning the bond strength of different MTA based materials as Biodentine, TheraCal, NeoMTA, and other earlier products to different restorative materials. However, the recently produced premixed bio- ceramic material "NuSmile- Neoputty" had not been sufficiently recognized in the dental literature, and did not take the same attention.

Research gap: To date, the shear bond strength of the newly introduced premixed bio- ceramic material NuSmile- Neoputty MTA to different restorative materials; especially those which are widely used in pediatric dentistry; hasn't been satisfactory investigated in the dental literature.

Aim of the study: The study was targeted towards assessment of micro-shear bond strength of four restorative materials that are widely used in pediatric dentistry to the premixed bio- ceramic MTA based material NuSmile- Neoputty. The four restorative materials were two glass ionomer restorative materials: Fuji II LC (Resin modified glass ionomer) and Equia Forte Fil (High viscosity glass ionomer). Also, two dental resin restorative composites: Cention N (Alkasite dental resin composite) and Venus Bulk Fill (Bulk fill dental resin composite). The effect of application of 10-Methacryloyloxydecyl dihydrogen phosphate (MDP) containing universal adhesive on the shear bond strength of NuSmile- Neoputty to the tested restorative materials was assessed in the study. Failure mode was analyzed throughout the course of the study. In addition, the effect of MDP containing universal adhesive on the surface of NuSmile-Neoputty was investigated through scanning electron microscopic examination.

Null hypotheses: The null hypothesis (1) claimed that no difference in the micro- shear bond strength of NuSmile- Neoputty MTA based material to the four tested restorative materials (Fuji II LC, Equia Forte Fil, Cention N, and Venus Bulk Fill) would be found. The null hypothesis (2) claimed that application of MDP containing universal adhesive would have no effect on the shear bond strength of NuSmile- Neoputty to the four tested restorative materials.

MATERIALS AND METHODS

Materials used in the study:

- **NuSmile- Neoputty** (Nusmile Inc., Houston, TX; USA). It is a premixed bio- ceramic mineral trioxide aggregate (MTA) based material. Described by the manufacturer as a bioactive

paste that consists of fine inorganic powder of tri-calcium/di-calcium silicate in a water- free organic medium. The working time at room temperature is about one hour. The initial setting time in- vivo at 37°C is approximately four hours (under the effect of moisture from the apical tissues, dentinal tubules or pulp tissue for setting). The material showed less than 3% solubility, which is considered an acceptable value by the ADA 57 [15,16], ISO 6876 [17] & ISO 9917-1[18].

- GC Fuji® II LC capsules (GC corp, Tokyo, Japan). It is a self adhesive light cured resin-modified glass ionomer. The capsule contains fluoro- alumino- silicate glass, 2-hydroxyl ethyl methacylate (HEMA), polybasic carboxylic acid (containing three carboxylic groups) and urethane di-methacrylate (UDMA), distilled water. The contents of the capsule are: 0.33 g powder; 0.085 ml liquid per capsule.
- Equia Forte Fil capsules (GC Corp., Tokyo, Japan). It is a high viscosity conventional glass ionomer. Each capsule contains powder and liquid in a ratio of 0.40 gm powder/0.10 ml liquid. The powder consists of ultra-thin (4μm-25μm) strontium- fluoro- alumino- silicate glass particles that are highly reactive, polyacrylic acid and iron oxide. The liquid constitutes of polybasic carboxylic acid and distilled water.

The Equia Coat consists of 50% methyl methacrylate and 0.09% camphorquinone.

- **Cention N** (Ivoclar Vivadent, Schaan, Liechtenstein.). An alkasite dual cured dental resin composite, of powder and liquid. The powder consists of fillers of approximately 57.6 % by volume calcium- barium- aluminium- fluoro- silicate glass, also iso-fillers comprising of cured di-methacrylates, ytterbium tri-fluoride. In addition to spherical mixed oxides are included to achieve desirable physico- mechanical properties. Initiators and pigments are included. The liquid contains di-methacrylates, initiators, stabilizers, and mint flavor.
- **Venus Bulk Fill** (Heraeus Kulzer, Germany). It is a single increment flowable bulk fill dental resin composite, which is composed of urethane di-methacrylate (UDMA) and ethoxylated-bisphenol A di-methylacrylate (EBADMA), tri-ethyleneglycol di-methacrylate (TEGDMA) matrix, and nano- hybrid fillers (38% by vol) of Ba- Al- Si glass,YbF_3, fumed SiO_2. It is supplied in simple dispensed syringes. It can be cured to a depth of 4mm.
- **Universal bonding adhesive** (3 M Single Bond universal adhesive, 3M ESPE, Deutschland GmbH, Neuss, Germany). It is composed of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer, di-methacrylate resins (Bis-GMA), HEMA, Vitrebond copolymer, fillers, ethanol, water, initiators and silane. Its pH equals 2.7. The pH was checked by a digital pH meter (Orion, 710 A, USA). It is considered to be an ultra- mild self- etch adhesive.

Sample size and specimens grouping

A total number of 164 specimens were used to perform the current study. 160 specimens were used for the shear bond strength testing and failure mode analysis. 80 specimens were performed with application of the universal adhesive, and other 80 specimens were performed without application of the universal adhesive. Four specimens were needed for scanning electron microscopic investigation of NuSmile- Neoputty material: two specimens were examined without application of the adhesive, and two specimens were examined after application of the universal. Sample size calculation was done using G Power version 3.1.9 at 90% power and 95% confidence interval. Specimens were divided into four groups (I, II, III & IV) according to the used restorative material bonded to NuSmile- Neoputty bio- ceramic material (40 specimens for each

group). Then each group was further subdivided into two subgroups (A&B) according to the universal adhesive application or not respectively (n= 20 for each subgroup).

Group I: Fuji II LC glass ionomer bonded to NuSmile- Neoputty.

IA: With application of the universal adhesive.

IB: Without application of the universal adhesive.

Group II: Equia Forte Fil glass ionomer bonded to NuSmile- Neoputty.

IIA: With application of the universal adhesive.

IIB: Without application of the universal adhesive.

Group III: Cention N dental resin composite bonded to NuSmile- Neoputty.

IIIA: With application of the universal adhesive.

IIIB: Without application of the universal adhesive.

Group IV: Venus Bulk Fill dental resin composite bonded to NuSmile- Neoputty.

IVA: With application of the universal adhesive.

IVB: Without application of the universal adhesive.

I- Shear bond strength assessment:

I.A - Preparation of NuSmile- Neoputty specimens.

Stainless steel molds having circular central holes of 2mm diameter and 2mm depth were used for specimens' preparation. The molds were fully filled with NuSmile- Neoputty material and condensed using a condenser, then covered with a microscopic glass slide and pressed by hand to obtain a cohesive specimen with flat surface. A load of 400 gm (copper bar) was put on top of the glass slide for 1 minute (Figure 1a); to standardize the final pressure on all specimens. After removal of the load, any excess material was carefully removed using a cement spatula. A wet cotton pellet that was soaked in water was put on top of the surface of the specimen and left for 10 minutes to initiate setting, and then specimens were left in an incubator (Titanox, ART.A 3213- 400 I, Italy) at 37 °C in 80% relative humidity for 7 days to ensure the complete setting of the Neoputty material, based on the delayed restorative technique applied in the current study.[3, 4, 19]

After the end of the storage period, specimens were removed from the incubator and the surface of each specimen was air- dried to be ready for the bonding procedures (Figure 1b).

I. B) Adhesive application and bonding procedures.

The universal adhesive was applied as one coat on the NuSmile- Neoputty material (without pre-etching)[3] using a micro- brush and rubbed for 20 seconds, then gently air dried for 5 seconds. Polyethylene tubes with an adhesive area of 0.8 mm^2 and 2mm in height (Tygon medical tubes, Saint Gobain, Lyon, France) were placed and centralized on top of the NuSmile- Neoputty surface.[20]

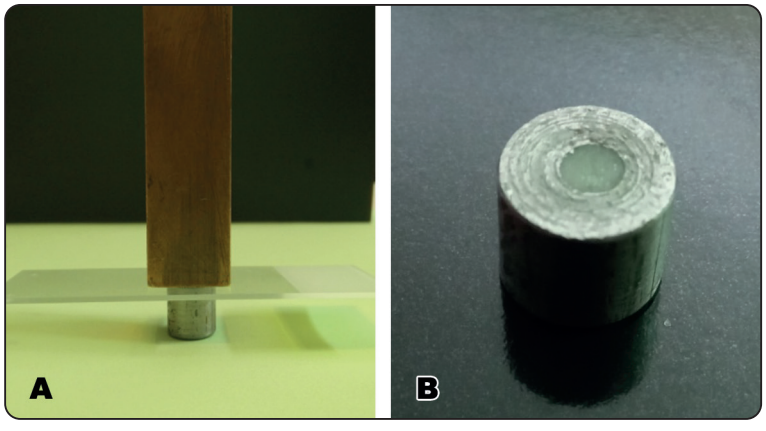


Fig. (1) a) A photo for a mold filled with the NuSmile- Neoputty material, covered with a microscopic glass slide and a load of 400 gm placed on top.

Fig. (1) b) A photo for a prepared NuSmile- Neoputty specimen within a stainless steel mold.

When the tubes were in place, the adhesive were light polymerized for 10 seconds, according to manufacturer instructions, using a LED light curing unit with an included light intensity sensor (Woodpecker LED.F, Guilin Woodpecker medical instruments Co., LTD), at light intensity of 1800 mW/ cm^2.

I. C) Preparation of restorative materials.

The restorative materials were prepared according to manufacturer instructions, as follows:

- **GC Fuji II LC:** The capsule was shaken to loosen the powder and then pressed and kept pressed for 2 seconds. The capsule was then transferred to an amalgamator (DMG, Hamburg, Germany, 4800 rpm) for mixing for 10 seconds. Then the capsule was loaded into the applier and clicked twice to prime the capsule before syringing the material. The material was then injected directly into the polyethylene tubes. Gentle pressure with a cement spatula was done to ensure having a compact specimen of no voids and obtaining a flat surface. Any excess material was removed with the spatula.
- Equia Forte Fil: Equia Forte Fil capsule was shaken to loosen the powder, and then pressed and kept pressed for 2 seconds. After that, the capsule was mixed by the amalgamator for 10 seconds. Then, the mixed capsule was loaded into the capsule applier; two clicks were performed to prime the capsule then injected into the polyethylene tubes. The excess of GIC was removed gently by a cement spatula, performing gentle pressure on the specimen surface to ensure adequate surface integrity and material compaction.[21]
- **Cention N:** Preparation of Cention N specimens' was achieved through hand mixing of two spoons of powder and 2 drops of liquid on a mixing pad. A plastic spatula was used for mixing till obtaining a smooth consistency, where the mixing time did not exceed one minute. The composite material was placed into the tubes using the cement spatula. The paste surface was pressed with the cement spatula to obtain a flat surface and ensure proper material compaction.
- **Venus Bulk Fill:** The flowable bulk fill dental resin composite was dispensed from the syringe and applied in the mold. While filling the mold, the tip of the syringe remained within the material during application to avoid entrapment of air voids.

After the tubes were filled with the dental restorative material used, and excess material was removed, specimens were then covered with a celluloid strip and light curing of the restorative material was performed according to manufacturer recommendations as following: 20 seconds for GC Fuji II LC, Cention N and Venus Bulk Fill. The curing tip of the LED curing unit was placed directly over the polyethylene tube and perpendicular to the specimen surface, touching the celluloid strip to standardize the curing distance. Concerning Equia Forte Fil, it was left for self curing for 5 minutes.

After completion of the restorative procedures, specimens were stored in distilled water at room temperature for 24 hours before being tested. After 24 hours, the tubes were carefully removed using a sharp scalpel. Any extra flashes of the restorative material extending beyond the base of the restorative specimen were also removed with a sharp blade[3] (Figure 2).

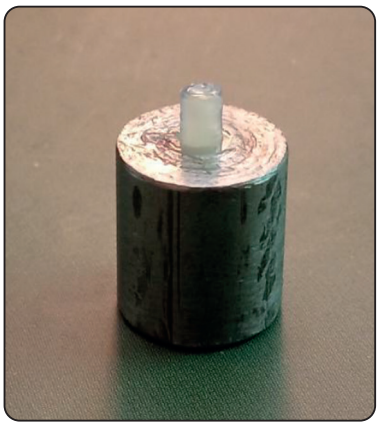


Fig. (2): A photo for bonded NuSmile-Neoputty/ Restorative material prepared for micro- shear bond strength testing.

I.D - Micro-shear bond strength testing.

For micro-shear bond strength (μSBS) testing, each stainless steel mold was secured to the lower jig of the universal testing machine (Instron Instruments 3365, USA, load cell of 5kN). Each specimen was tested for micro-shear bond strength using a chisel load applicator of 0.3 mm tip thickness; which was placed at the NuSmile- Neoputty/ restorative material interface, precisely guided by the NuSmile- Neoputty surface. The test was run at a cross head speed of 1 mm/ minute until debonding had occurred. The force required to debond the resin composite was measured in Newton (N). The SBS was calculated by dividing this load (force) by the area of the bonding interface in mm^2 and measured in MPa.[4, 22]

II- Assessment of failure mode

The fracture surfaces were evaluated under a stereomicroscope (Olympus – Japan) at a 24X magnification. The magnification is the product of the adjustment of the objective lens (1.2X) × the camera eye piece enlargement (20X).

The fracture pattern was classified as follows:

- ***Adhesive failure***: denoting failure between the NuSmile- Neoputty material and the restorative material with no remnants on any of the materials surfaces.
- ***Cohesive failure type 1***: failure occurred within the NuSmile- Neoputty bio- ceramic material.
- ***Cohesive failure type 2***: failure occurred within the restorative material.
- ***Mixed failure type 1***: denoting both adhesive and cohesive failure of type 1.
- ***Mixed failure type 2***: denoting both adhesive and cohesive failure of type 2.

The debonded surfaces of the restorative materials were also inspected.

III- Assessment of NuSmile- Neoputty surface after application of the universal adhesive: Scanning electron microscopic examination.

The surface of NuSmile- Neoputty material was assessed by scanning electron microscope (SEM); without application of the universal adhesive and also after application of the universal adhesive. A total of four specimens were prepared in a split Teflon mold of square shaped hole of 2mm sides and thickness. The molds were fully filled with the material and then covered with a glass slide and gently finger pressed. This procedure was performed to standardize the smoothness of the specimens' surfaces. Excess material was then removed, and a wet cotton pellet was put on top of the surface of the specimen and left for 10 minutes to initiate setting. Specimens were then left for complete setting, and also to follow the delayed restoration technique performed in the study. Specimens were kept in the incubator at 37 °C in 80% relative humidity for 7 days, as in section I.A. After that, for the two specimens concerning application of the universal adhesive, the adhesive was applied on the top surface of specimens, according to manufacturer instructions, as mentioned in section I- B. Specimens were then ready for SEM examination. Specimens were mounted on aluminum tubs. Gold sputtering was performed to the surface treated with the adhesive; using a gold sputter coater: Polaron, SC: 7620, UK. Enamel surfaces were then observed under SEM: JSM 6060 (JEOL), Japan, at a magnification of (2500X).

Statistical analysis:

Categorical data were presented as frequency and percentage values and were analyzed using chi-square test followed by pairwise comparisons utilizing multiple z-tests with Bonferroni correction. Numerical data was represented as mean and standard deviation (SD) values.Shapiro-Wilk's test was used to test for normality. Homogeneity of variances was tested using Levene's test. Data showed parametric distribution and variance homogeneity and were

analyzed using two-way ANOVA followed by Tukey's post hoc test. Comparison of simple main effects was done utilizing the error term of the two-way model with p-values adjustment using Bonferroni correction. The significance level was set at $p<0.05$ within all tests. Statistical analysis was performed with R statistical analysis software version 4.1.3 for Windows (R Core Team (2022). R: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria).

RESULTS

I- Results of micro- shear bond strength

Results of two-way ANOVA for micro-shear bond strength values presented in table (1), showed there was a significant interaction between type of restorative material and adhesive application ($p<0.001$).

Comparison of simple main effects presented in table (2) showed that for subgroup (A), there was a significant difference between tested groups ($p<0.001$). Post hoc pairwise comparisons showed that groups I (18.33±2.29 MPa) and III (18.52±2.63 MPa) had revealed significantly higher values than other groups ($p<0.001$), with no significant difference between them. In addition, group IV (12.96±1.76 MPa) revealed significantly higher shear bond strength value than group II ($p<0.001$). Group II (7.07±1.06 MPa) yielded the lowest shear bond strength value among all subgroups A.

For subgroup (B), the difference was not statistically significant ($p=0.999$). For group (II), there was no significant difference between subgroups (A) and (B) ($p=0.998$), while for other groups, subgroups (A) had significantly higher shear bond strength value than subgroup (B) ($p<0.001$).

Mean and standard deviation values for micro-shear bond strength in different groups were presented in figures (3) and (4).

Table (1):Two-way ANOVA test results

Parameter	*Sum of squares*	*df*	*Mean square*	*f-value*	*p-value*
Restorative material	872.75	3	290.92	100.14	**<0.001***
Adhesive application	2064.61	1	2064.61	710.66	**<0.001***
Material*Adhesive	890.45	3	296.82	102.17	**<0.001***
Error	441.59	152	2.91		

significant (p<0.05)

Table (2): Comparisons of simple main effects

Material / *Adhesive*	*Micro-shear bond strength (MPa) (Mean±SD)*				*f-value*	*p-value*
	Group (I)	*Group (II)*	*Group (III)*	*Group (IV)*		
Subgroup (A)	18.33 ± 2.29^{A}	7.07 ± 1.06^{C}	18.52 ± 2.63^{A}	12.96 ± 1.76^{B}	**202.30**	**<0.001***
Subgroup (B)	7.02 ± 1.12^{A}	7.07 ± 1.45^{A}	7.00 ± 1.37^{A}	7.05 ± 1.27^{A}	**0.01**	**0.999**
f-value	**439.80**	**0.00**	**457.04**	**120.33**		
p-value	**<0.001***	**0.998**	**<0.001***	**<0.001***		

Different superscript letters indicate a statistically significant difference within the same horizontal row; *significant (p<0.05).

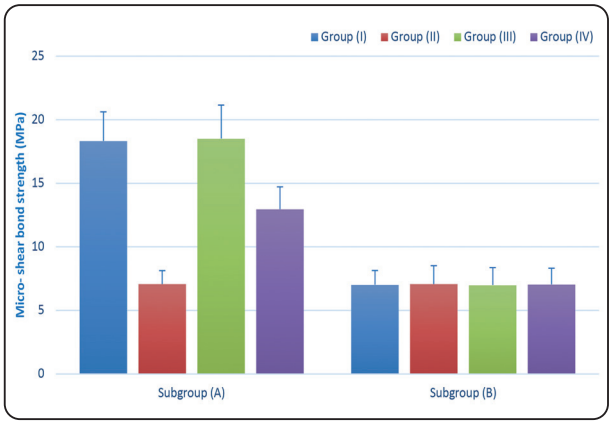


Fig. (3): Bar chart showing mean and standard deviation values of micro-shear bond strength (MPa) values in different groups in relevance to the tested restorative material.

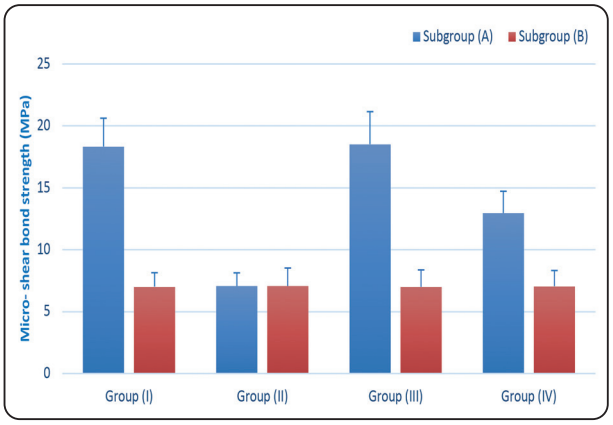


Fig. (4): Bar chart showing mean and standard deviation values of micro-shear bond strength (MPa) values in different groups in relevance to adhesive application.

II- Results of failure mode

Stereomicroscopic photos representing different failure modes were represented in figures (5&6); with magnification 24X. Failure mode results were statistically shown in tables (3 & 4) and represented as bar charts in figures (7 & 8).

i) Specimens involving application of the universal adhesive (subgroups IA, IIA, IIIA & IVA):

Mixed failure of type 1 was found in all specimens **(100%)** of subgroups **IA** and **IIIA**. Areas of adhesive failure were seen, and others showing cohesive failure within the NuSmile- Neoputty material, leaving deficient areas on its surface (white arrow) (Figure 5a). A stereomicroscopic photo for the debonded restorative material surface is shown in figure (6a) showing parts of NuSmile- Neoputty material attached to the restorative material surface (white arrow).

Subgroup IIA showed **adhesive failure** in all specimens **(100%)**. A stereomicroscopic photo for the NuSmile- Neoputty surface is shown in figure (5b), and a stereomicroscopic photo for the debonded Equia Forte Fil restorative material surface is shown in figure (6b).

Subgroup IVA showed **mixed failure type 2** in 17 specimens **(85%)**. By examining the debonded surface of NuSmile- Neoputty material; areas of adhesive failure were shown (yellow arrow), and areas of cohesive failure were found within the composite material (Red arrow), where remnants of Venus composite were seen attached to the NuSmile-Neoputty surface (Figure 5c). The debonded Venus Bulk Fill surface is shown in figure (6c); a deficient area in composite due to cohesive failure was revealed (red arrow), and area of adhesive failure was seen (yellow arrow). Subgroup IVA also yielded 3 specimens **(15%)** showing **adhesive failure**.

ii) Specimens without application of the universal adhesive (subgroups IB, IIB, IIIB & IVB):

All specimens of all groups showed **adhesive failure (100%).**

iii) Results of scanning electron microscopic examination

The surface of specimens that were subjected to application of MDP containing universal adhesive was relatively more irregular and showed more surface micro- porosities (Figure 9b) in comparison to specimens that were not subjected to the universal adhesive application (Figure 9a).

TABLE (3): Effect of restorative material on failure mode

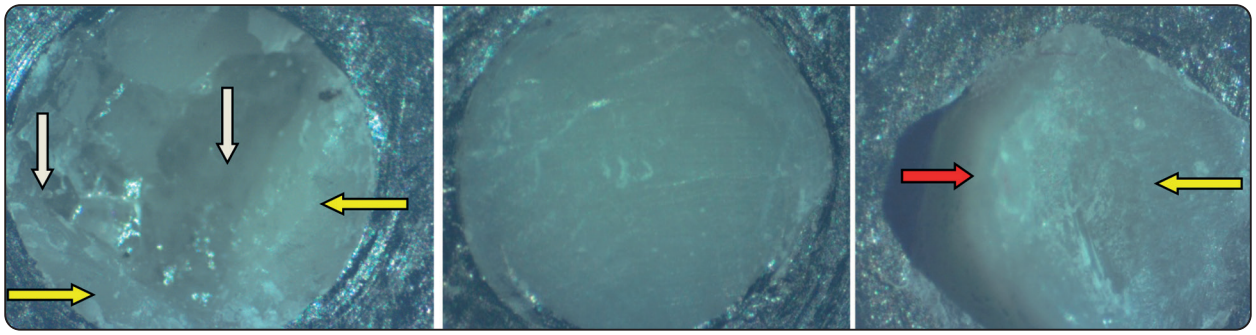
Material Adhesive	*Failure mode*	*Group (I)*		*Group (II)*		*Group (III)*		*Group (IV)*		χ^2	*p-value*
		n	**%**	**n**	**%**	**n**	**%**	**n**	**%**		
Subgroup (A)	*Adhesive failure*	0^A	0.0%	20^B	100.0%	0^A	0.0%	3^A	15.0%	**139.13**	**<0.001***
	Cohesive failure type 1	0^A	0.0%	0^A	0.0%	0^A	0.0%	0^A	0.0%		
	Cohesive failure type 2	0^A	0.0%	0^A	0.0%	0^A	0.0%	0^A	0.0%		
	Mixed failure type 1	20^A	100.0%	0^B	0.0%	20^A	100.0%	0^B	0.0%		
	Mixed failure type 2	0^A	0.0%	0^A	0.0%	0^A	0.0%	17^B	85.0%		
Subgroup (B)	*Adhesive failure*	20	100.0%	20	100.0%	20	100.0%	20	100.0%	**NA**	**NA**
	Cohesive failure type 1	0	0.0%	0	0.0%	0	0.0%	0	0.0%		
	Cohesive failure type 2	0	0.0%	0	0.0%	0	0.0%	0	0.0%		
	Mixed failure type 1	0	0.0%	0	0.0%	0	0.0%	0	0.0%		
	Mixed failure type 2	0	0.0%	0	0.0%	0	0.0%	0	0.0%		

Different superscript letters indicate a statistically significant difference within the same horizontal row; *significant (p<0.05).

TABLE (4): Effect of adhesive application on failure modes.

Adhesive Material	*Failure mode*	*Subgroup (A)*		*Subgroup (B)*		χ^2	*p-value*
		n	**%**	**n**	**%**		
Group (I)	*Adhesive failure*	0^A	0.0%	20^B	100.0%	**40.00**	**<0.001***
	Cohesive failure type 1	0^A	0.0%	0^A	0.0%		
	Cohesive failure type 2	0^A	0.0%	0^A	0.0%		
	Mixed failure type 1	20^A	100.0%	0^B	0.0%		
	Mixed failure type 2	0^A	0.0%	0^A	0.0%		
Group (II)	*Adhesive failure*	20	100.0%	20	100.0%	**NA**	**NA**
	Cohesive failure type 1	0	0.0%	0	0.0%		
	Cohesive failure type 2	0	0.0%	0	0.0%		
	Mixed failure type 1	0	0.0%	0	0.0%		
	Mixed failure type 2	0	0.0%	0	0.0%		
Group (III)	*Adhesive failure*	0^A	0.0%	20^B	100.0%	**40.00**	**<0.001***
	Cohesive failure type 1	0^A	0.0%	0^A	0.0%		
	Cohesive failure type 2	0^A	0.0%	0^A	0.0%		
	Mixed failure type 1	20^A	100.0%	0^B	0.0%		
	Mixed failure type 2	0^A	0.0%	0^A	0.0%		
Group (IV)	*Adhesive failure*	3^A	15.0%	20^B	100.0%	**29.57**	**<0.001***
	Cohesive failure type 1	0^A	0.0%	0^A	0.0%		
	Cohesive failure type 2	0^A	0.0%	0^A	0.0%		
	Mixed failure type 1	0^A	0.0%	0^A	0.0%		
	Mixed failure type 2	17^A	85.0%	0^B	0.0%		

Different superscript letters indicate a statistically significant difference within the same horizontal row; *significant (p<0.05).

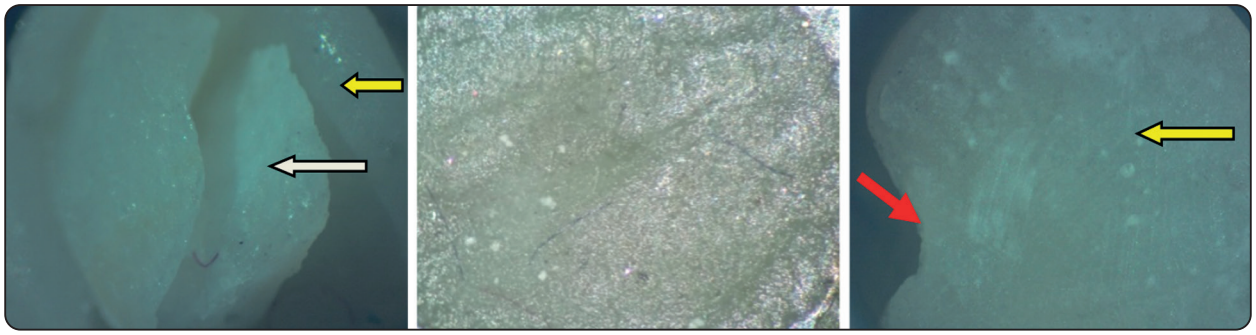


a: Mixed failure type 1 Adesive failure (yellow arrow). Cohesive failure within the NuSmile- Neoputty material leaving deficient area (white arrow).

b: Adhesive failure

c: Mixed failure type 2. Adhesive failure (yellow arrow). Cohesive failure within Venus composite (red arrow).Venus composite material attached to the NuSmile-Neoputty surface

Fig. (5): Stereomicroscopic photos for NuSmile- Neoputty surfaces (24X) representing failure modes in specimens of the tested groups.



a: Cention N composite surface after debonding showing parts of NuSmile-Neoputty material attached to it, representing mixed failure type 1. Adesive failure (yellow arrow). Cohesive failure within the NuSmile- Neoputty material (white arrow).

b: Equia Forte Fil surface after debonding showing adhesive failure.

c: Venus Bulk Fill composite specimen after failure. The debnded surface showed mixed failure type 2. Adhesive failure (yellow arrow). Cohesive failure within the composite material (red arrow).

Fig. 6: Stereomicroscopic photos for the restorative materials surfaces (24X) representing failures in different groups.

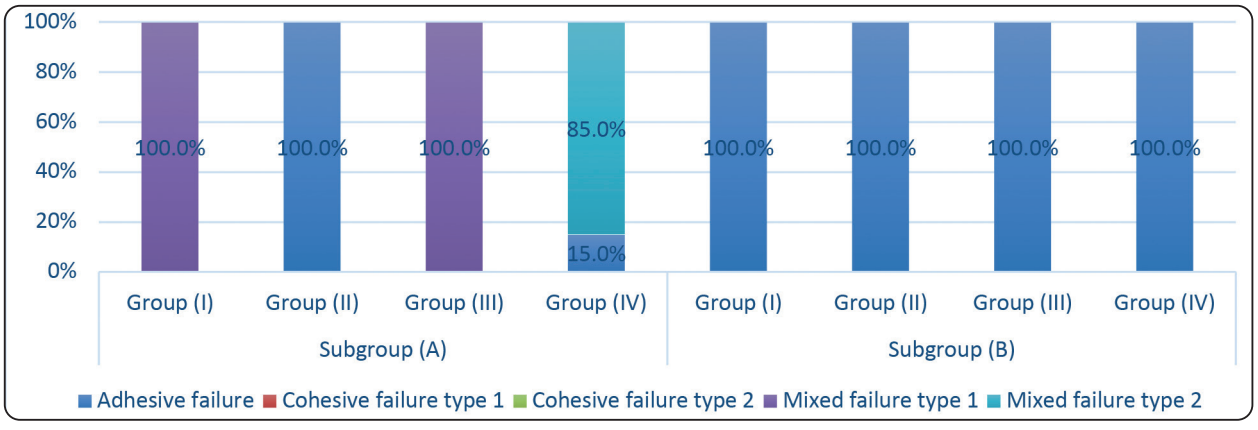


Fig. (7) Stacked bar chart showing effect of restorative material on failure mode.

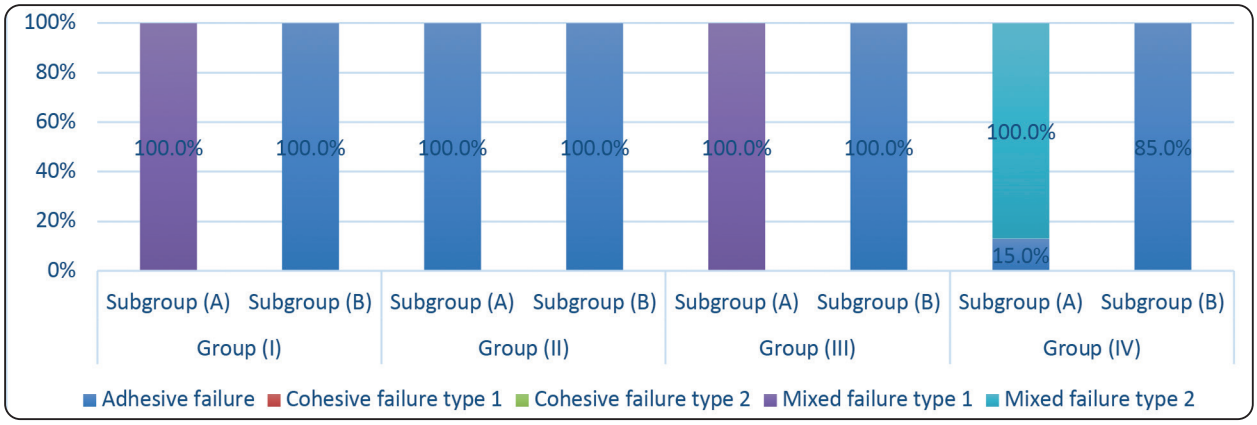


Fig. (8) Stacked bar chart showing effect of adhesive application on failure mode.

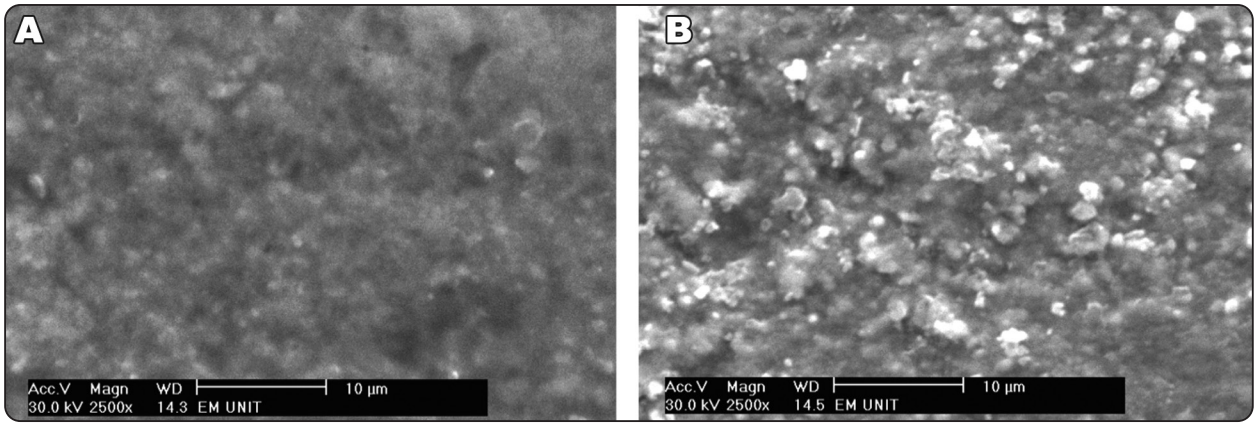


Fig. Fig. (9) a: Scanning electron microscopic photomicrograph for NuSmile- Neoputty surface without application of the universal adhesive. A relatively more regular surface in comparison to the etched surface. Fig. 9b: Scanning electron microscopic photomicrograph for NuSmile- Neoputty surface after application of the universal adhesive. A more irregular surface was detected, with more surface micro- porosities.

DISCUSSION

Pediatric dentistry is considered a highly precise specialty that provides a comprehensive dental care for a critical age range patients. Dentists who are specialized in pedodontics require a continuous and immense focus on all new dental materials that are suitable for pediatric dentistry, as well as the new treatment modalities; in order to provide their dental service in skillful and professional approach.

The past few years showed new developments for more suitable restorative materials for pediatric patients. There are many options available to pedodontists for restoring primary teeth. Variable factors can guide the choice of pediatric restorations as the child's age, the caries index, the degree of cooperation of the child, type of the tooth to be treated and last but not least the type of material to be used. Meticulous choice of the material and knowing the precise manipulation steps and tricks is mandatory to avoid further tooth destruction, and to render the tooth/restoration interface caries resistant. Furthermore, to select a material that can withstand the tremendous oral environment for the time duration in which it is in service.[23]

Obtaining a clear understanding of the unique properties and points of weakness and strengths

of the available materials made it crucial to assess the premixed bioactive bio- ceramic MTA material NuSmile- Neoputty, which is recently used by pediatric dentists for pulp related treatments. NuSmile gained great attention due to many privileges compared to other MTA based materials. The concentration of the bioactive powder in NuSmile was increased and also delivered in a formula that allows fast hydration reaction and formation of Ca $(OH)_2$ needed for hydroxyapatite formation. Previously developed light-cure and dual-cure MTA products contain resins that lessen the MTA's bioactivity; as it hinders the hydration reaction. Also, resins never achieve complete 100 % curing, leaving residual uncured resin that can induce pulpal irritation. Additionally, resins shrink during curing, jeopardizing the material/ tooth adhesive junction. On the other hand, NuSmile-Neoputty doesn't contain resinous material; offering a high degree of hydration reaction and bioactivity. In relevance to dimensional changes; it shows minor setting expansion that offers good sealing quality.[24]

Assessment of the bond strength of NuSmile-Neoputty to the restorative materials (Fuji II LC, Equia Forte Fil, Cention N, and Venus Bulk Fill) was of crucial importance due to the wide range of use of these restorative materials in the field of pediatric dentistry.

Attaining adequate bond strength between calcium silicate- based materials and restorative materials; either glass ionomer or dental resin composites; is one of the main keystones for achieving a successful restoration. High shear bond strength can be considered a pivot for providing favorable adhesion and enhanced retention of the restoration. Also, the higher shear bond strength leads to decreased microleakage, and consequently can attain a restoration of gap- free margins.[25]

Shear bond strength can be tested either macro-shear or micro- shear testing. Micro- shear is more recommended as it reveals more accurate results, since reducing the testing area reduces the structural defects. For that reason, the present study was conducted through applying micro- shear bond strength testing.[22]

Shear bond strength testing can be conducted by using a knife edge chisel, wire loop or push out tests. It was previously revealed that using the wire loop method cannot guaranty a 100% highly precise shear bond strength values. The authors attributed this result to application of load at a distance from the adhesive junction interface. This distance is caused by the wire cross section, so that the specimen loading occurs by bending rather than by shearing stresses.[26] The bending that occurs most probably results in decreasing the force required for failure at the adhesive junction, and so decreases the stress at failure.[22] On a practical basis, the wire loop can show a possibility of slippage during testing. This can adversely affect the reliability of the testing method; since some of the loading force can be lost on disrupting the cohesive forces of the substrate material, rather than shearing the adhesive interface itself. As a consequent, the resultant values may not precisely represent the strength of the adhesive interface, but also can include the cohesive strength of the substrate material.[26] Based on this information; no sharp preference could be suggested for either techniques of load application (the wire loop or the chisel). It was known that the knife-edged chisel was the traditional loading method proposed by ISO/TR 11405:1994[27] despite concerns regarding stress concentration at a specific point on the bonded interface, leading to complex representation of stresses and underestimated bond strength value. However, it is applicable to place the thin knife edge of the chisel at the bonded interface, and the chisel can be guided by the flat surface of the substrate to concentrate the stresses at the bonded interface.[22] Using a very thin knife- edge could be also helpful to reach a consistent testing technique. For these reasons, the knife- edge chisel technique was chosen in the current study.

The manufacturer claimed that performing the desired restoration or cementing a crown can be performed immediately after placing the NuSmile-Neoputty material. However, several studies had advised to delay the restorative procedures for 72 to 96 hours after NuSmile- Neoputty placement, in order to permit the material to reach complete setting and achieve its optimum mechanical properties. A delay for 7 days was also recommended in some studies to avoid degradation of the material by the dental procedures including etching, rinsing, and priming.[4, 28, 29] Alqahtani et al. in their study in 2022[4] had confirmed that the 7 days delay in performing the restorations by either dental resin composite (Filtek Z350 XT flowable composite using single bond universal adhesive 3M), or resin modified glass ionomer (GC Fuji II LC) after the premixed bio-ceramic material (Neoputty) placement had shown higher shear bond strength values in comparison to immediate restorations.

Regarding application of conventional glass ionomer on top of MTA based materials, previous investigations[28, 29] had supported the delayed restoration after MTA material placement. It was claimed that glass ionomer was responsible for water sorption from the freshly mixed MTA material. This had lead to incomplete hydration of MTA, as well as occurrence of remarkable porosity. The interface junction between glass ionomer and MTA exhibited a high degree of micro- cracking, with the two materials getting away from each other, resulting in deteriorated adhesive junction.

Concerning resin modified glass ionomer, Alqahtani et al.[4] had mentioned that when Fuji II LC resin modified glass ionomer was immediately placed on ProRoot MTA it revealed statistically higher shear bond strength than the delayed placement. Several studies[4, 28- 31] had correlated the relatively strong bond of resin modified glass ionomer to the unset MTA based material could be related to the ability of formation of chemical bonding between the carboxylate anions (RCOO–) in the polyacrylic acid and the calcium in the MTA during its setting. On the contrary, those studies had declared that the premixed bio- ceramic (Neoputty) had responded in a contradictory way to either ProRoot MTA or NeoMTA2 concerning the immediately placed resin modified glass ionomer; as Neoputty had revealed lower shear bond strength values on being immediately restored compared to the delayed restoring. This was attributed to the need of the premixed bio- ceramic Neoputty for moisture from external sources to start the setting reaction.[4,28-31] Thus, when dealing with premixed bio- ceramics it was found not preferable to immediately place the resin modified glass ionomer on top. Consequently, in accordance to the recommendations of the previously conducted researches, the present study went through the delayed restoration technique, and NuSmile- Neoputty specimens were left stored for 7 days before placement of the restorative materials.

The 7 days delay was done through storage of NuSmile- Neoputty specimens in an incubator at 80% relative humidity before restoration construction. The storage was performed in 80% not 100% since the 100% relative humidity was not adjustable with the required storage temperature (37^{O}C) in the used incubator (Titanox), because a decrease of the relative humidity takes place to about 50% to 60% as the temperature increases at a range from 40^{O}C to 50^{O}C.

Concerning surface amendment of MTA based materials prior to restorative materials application; investigations were performed regarding acid etching the MTA based material, and also using an adhesive in order to augment their bonding to dental resin composite restorations. In some studies[32- 37] it was assumed that acid etching can induce higher shear bond strength of resin composite with some MTA based materials. Those studies revealed that acid etching with phosphoric acid can alter the MTA material surface and create a surface with micro-

porosities, enhancing micromechanical interlocking with the bonding resin. On the contrary, several authors [38- 41] had previously revealed an opposite thought; that acid etching as a separate step can induce MTA surface degradation, reduction in the cohesive strength, deteriorated MTA micro-hardness, decreased compressive strength, as well as degradation of MTA and formation of an amorphous gel- like surface structure, with removal of the desirable needle-shaped crystals that are useful for bonding with the resin. These changes can consequently induce an adverse effect on the shear bond strength of the MTA material to the dental resin composite restoration. Also, the need for simplifying the dental procedures and reduce the working time is of prime importance when dealing with pediatrics. In line with this information, it could be deduced that the use of a universal bonding agent without a separate pre- etching step would be preferred. Additionally, the choice of 3 M Single Bond universal adhesive to be used in the current study was based on its pH 2.7; which made it considered an ultra- mild etching system[42,43] and so can provide suitable surface micro- irregularities of the Neoputty surface without jeopardizing the surface crystals needed for creating a strong adhesive junction.

For micromechanical interlocking, using functional monomers can be beneficial. Functional monomers have two functional groups; one group that can etch the MTA surface and another that is capable for polymerization.[29, 41, 44,45] 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer is a functional acidic monomer; where its acidity was relied upon to etch the MTA surface, and so can facilitate the resin penetration for producing mechanical interlocking adhesion. In addition, if the adhesive has low viscosity, this can help its easier penetration into the MTA cement. Furthermore, the addition of 10- MDP as a constituent in the adhesive system may facilitate the chemical "chelation" interaction with the calcium rich MTA surface, creating chemical bonding.[29] Despite the wet condition of the NuSmile- Neoputty material, bonding to the applied universal adhesive could be considered possible due to the structural formula of the 3M single bond universal adhesive. Vitrebond Copolymer and HEMA in the adhesive can help proper wetting of the NuSmile surface and so enhance bonding.

Regarding glass ionomer restorations, both chemical and micromechanical adhesion were suggested as mechanisms for bonding to tri-calcium silicate-based materials.[28] Micromechanical interlocking can be achieved by the etching effect of the acidic MDP monomers in the universal adhesive. Chemical bonding can be based on the interaction of (COO^-) groups in glass ionomer and calcium in MTA based materials. But it must be considered that this adhesive junction is liable for hydrolysis; so presence of MDP containing adhesive can be beneficial, as it can offer a possibility of formation of stable (water insoluble) interaction between MDP molecules in the adhesive and Ca salts in the MTA material by chemical chelation. The 10-MDP monomer has long hydrophobic chains that can produce a rich MDP/ Ca salt adhesive interface of strong hydrophobicity [46], which improves the adhesive junction strength and increases its resistance to hydrolysis. As a consequent, it can strengthen the glass ionomer/ MTA bond.[44,45] In the light of those earlier findings, a universal adhesive containing 10- MDP was used in the present study in order to assess its effect on the strength of the adhesive junction between NuSmile- Neoputty to the tested restorative materials and clarify whether it can enhance the bond strength or not.

Based on the results obtained from the present research, the null hypothesis 1 was partially rejected; since the μSBS of GC Fuji II LC (subgroup IA) and Cention N (subgroup IIIA) to NuSmile-Neoputty material was statistically comparable. However, Venus Bulk Fill (subgroup IVA) revealed

statistically significant lower shear bond strength to NuSmile- Neoputty compared to subgroups IA and IIIA. The least shear bond strength values among the tested groups were revealed by Equia Forte Fil (subgroup IIA). Concerning the effect of application of the universal adhesive on the bond strength; the null hypothesis 2 was partially rejected. The application of the universal adhesive had resulted in a significant increase in the shear bond strength in the tested groups I, III & IV. However, group II showed no significant difference.

The ultra- mild etching potential of the universal bonding agent (due to its pH 2.7) can be relied upon on creating surface micro- irregularities on the Neoputty surface; which could be considered the key for inducing micromechanical interlocking of the restorative materials with the Neoputty surface. This micromechanical interlocking adhesive mechanism offered by the universal adhesive could be considered a common bonding mechanism in all tested groups. Scanning electron photomicrograph in figure (9b) showed the surface irregularities and micro-porosities of NuSmile- Neoputty material after application of the MDP containing universal adhesive.

The highest shear bond strength among the tested groups involving application of the universal adhesive was for subgroup IA, where GC Fuji II LC was bonded to NuSmile- Neoputty (18.33±2.29 MPa). This result can be attributed not only to the micromechanical interlocking with the Neoputty material, but can be more attributed to the chemical bonding that can take place between the resinous component in the resin modified glass ionomer Fuji II LC with the applied adhesive, through the di-methacrylates and HEMA constituents in both materials, in addition to the silane constituent in the universal adhesive that can induce chemical bonding to silicates in the glass ionomer, and on the other side in the NuSmile-Neoputty MTA material. Also, the universal adhesive has the ability of formation of a stable MDP/ Ca salt adhesive interface with the NuSmile- Neoputty; since the MDP molecules in the universal adhesive were claimed to be capable of bonding to calcium in Neoputty MTA material, as that formed with calcium in the tooth structure.[44, 45] This can induce a strong Fuji II LC glass ionomer/ Neoputty MTA adhesive junction.

On comparing the results of the present study to a previous study conducted by Tulumbaci et al. in 2017[25] that measured the shear bond strength of resin reinforced glass ionomer (Photac Fil) to white MTA material and Biodentine without using an adhesive, their study yielded shear bond strength of values of (2.84 MPa and 2.59 MPa) respectively. Also, Alqahtani et al. in 2022[4] had assessed the shear bond strength of Fuji II LC to Neoputty material, without an adhesive application, and revealed a mean value of (1.62±0.12MPa). The difference in shear bond strength values between the present study and those previously conducted studies could emphasize that applying the MDP containing universal adhesive to NuSmile- Neoputty can enhance its bond strength to Fuji II LC resin reinforced glass ionomer, especially if the adhesive contains polyalkenoic acid. The results of those previously conducted studies were in line and can support the results of the present study that showed a significant increase in the shear bond strength values of subgroup IA (18.33±2.29 MPa) compared to subgroup IB (7.02±1.12 MPa), as presented in table (2) and figure (4), clarifying the beneficial effect of applying the MDP containing universal adhesive on the strength of the adhesive junction.

Concerning subgroup IIIA (Cention N bonded to NuSmile- Neoputty); it yielded a shear bond strength value of (18.52±2.63 MPa). Subgroup IIIA showed no statistical significant difference when compared to subgroup IA. The strong adhesive junction could be reliant on the chemical bonding with the adhesive through the resinous constituents (di-methacrylates) in both materials. Also another

explanation can be proposed based on the possibility of chemical bonding to take place between the MDP molecules in the universal adhesive with calcium in both the Neoputty material and Cention N composite, resulting in formation of a strong stable MDP/ Ca adhesive junction.[47,48] This chemical bonding mechanism could be considered a pivot for the strong Neoputty/ Cention N adhesive junction, in addition to the micromechanical interlocking mechanism induced by etching the Neoputty surface.

Application of the universal adhesive had enhanced the bond strength and this was revealed on comparing the results of subgroup IIIA (18.52±2.63 MPa) to subgroup IIIB (7.00±1.37 MPa), presented in table (2) and figure (4).

Venus Bulk Fill flowable composite, with application of the adhesive (subgroup IVA) revealed shear bond strength value of (12.96±1.76 MPa). The bonding mechanism of Venus composite to Neoputty material could be attributed to the chemical interaction of the resinous di-methacrylate groups in both the composite material and the adhesive. It could be attributed as well to the relatively low viscosity of the flowable composite that might have enhanced its penetration potential in the Neoputty surface micro- irregularities, inducing a strong adhesive junction. A possible explanation for the lower shear bond strength shown in subgroup IVA (Venus Bulk Fill) in comparison to groups IA (Fuji II LC) and IIIA (Cention N) could rely on the cohesive strength of the flowable composite itself; being a diffusible material forming a part of the adhesive junction. The relatively low filler content of Venus Bulk Fill flowable composite (38% by vol.) might have rendered it of low flexural strength[49], which might have been negatively reflected on the strength of adhesive junction as a whole. In contrast, Cention N with its thick polymer network and high degree of polymerization[11], as well as the filler percentage (57.6% by vol), along with presence of iso- fillers constituting 17% by volume[50], and presence of spherical mixed oxides could be considered responsible for providing a stronger adhesive junction in comparison to Venus Bulk Fill.

Concerning Fuji II LC, the reason behind its higher shear bond strength to NuSmile- Neoputty compared to that revealed by Venus Bulk Fill could be related to the polybasic carboxylic acid found in Fuji II LC. This Polybasic carboxylic acid can possibly induce chemical integration with the polyalkenoic acid in the universal adhesive, resulting in enhanced bonding to calcium in the NuSmile- Neoputty material.

On comparing the shear bond strength result of subgroup IVA (12.96±1.76 MPa) to that of subgroup IVB (7.05±1.27 MPa) could clarify that application of MDP containing universal adhesive had significantly enhanced the bond strength. This could be attributed mainly to the micro-porosities and etching of the NuSmile- Neoputty surface by the action of the universal adhesive, helping the micro-mechanical interlocking bonding mechanism.

The lowest shear bond strength value among the groups involving adhesive application was given by subgroup IIA (7.07±1.06 MPa). Equia Forte Fil is a high viscosity conventional glass ionomer. No resinous components were included within the composition of Equia Forte; consequently no significant chemical bonding could be created with the resinous component of the universal adhesive. Also, by studying the chemical composition of Equia Forte Fil; it was found that strontium substituted the presence of calcium in the glass particles; indicating the questionable capability of chemical bonding with MDP molecules. As a result, it could be called into attention that Equia Forte Fil might not be able to form a considerable chemical bonding with the universal adhesive, leading to a weak adhesive junction. The only way of bonding in this group could be mainly the micromechanical interlocking induced by etching the Neoputty

surface with the ultra- mild universal adhesive. However, even this micromechanical interlocking could not be considered of significant effect in this case; due to the high viscosity of the Equia Forte that might decrease its flow and penetration within the Neoputty surface micro-irregularities, specially that no chemical bonding was found to be created between the adhesive and Equia Forte Fil. A previously study conducted by Duman et al., in 2021[51] had tested the shear bond strength of Equia Forte Fil to NeoMTA and other calcium silicates without using an adhesive, and revealed bond strength of (3.60±2.46 MPa) between Equia Forte Fil and NeoMTA; which is considered a low bond strength value. NeoMTA could be considered comparable to NuSmile- Neoputty in the main compositional constituents. Thus, when comparing the results of the present study to the study conducted by Duman et al.[51], it could be emphasized that using MDP containing adhesives in case of dealing with Equia Forte Fil would show no benefit for its bonding potential to NuSmile- Neoputty. The results of Duman et al., in 2021[51] was found in agreement with the results of the present study; since both subgroups IIA (7.07±1.06 MPa) and IIB (7.07±1.45 MPa) revealed a statistically comparable shear bond strength values of no significant difference between them. Additionally, on focusing on the composition of Equia Forte Fil, the lack of calcium which is substituted by strontium could be a cause of weak bonding to NuSmile- Neoputty, even in case of direct application without an intervening adhesive layer, clarifying that presence of MDP containing adhesive or its absence would not affect the adhesive junction strength in case of bonding to Equia Forte Fil.

Regarding the failure mode assessment; it can't be considered a fundamental criterion for judging the success of the adhesive procedure, whereas the ultimate strength of the adhesive junction is a more important and more reliable.[3] Still, assessment of failure pattern could be performed in adjunct to the bond strength testing in a trial to obtain a more comprehensive evaluation of the adhesion quality. Concerning the present study; the classification of failure mode was done according to the pilot study results. The results obtained from the present study (tables 3 & 4, figures 7 & 8) had revealed mixed failure of type 1 in all specimens of subgroups IA and IIIA. Stereomicroscopic examination showed that areas showing cohesive failure within the bio-ceramic material had the greater percentage over the areas showing adhesive failure, going in line with their high bond strength values. For subgroup IIA, the failure mode (figures 5 & 6) was adhesive, supporting the weak penetration potential of Equia Forte within the etched bio- ceramic surface, and going in line with its least bond strength values among the tested materials. However, for subgroup IVA the failure was mixed type 2 in most of the specimens (85%), where the areas showing cohesive failure were within the composite resin material. This finding could be a reflection for the weak flexural properties of Venus Bulk Fil flowable composite[49], and goes in line with the shear bond strength results.

Regarding specimens performed without application of the universal adhesive; the type of failure was adhesive failure in all tested groups, revealing a weak and compromised adhesive junction. This was supported by the scanning electron microscopic examination that revealed the un- etched NuSmile-Neoputty surface showing relatively fewer irregularities and less micro- porosities in relation to the etched surface (Fig. 9). As a consequence, it could be detected that the un-etched NuSmile- Neoputty surface was found less capable for providing micro-mechanical interlocking adhesion compared to the etched surface. This obtained information consequently clarifies the importance of universal adhesive application, particularly on dealing with Fuji II LC, Cention N, and Venus Bulk Fill restorative materials.

The present study had covered an important aspect regarding the new premixed bio-ceramic material "NuSmile- Neoputty", which is its bond strength to restorative materials that have shown wide range of use in pediatrics. The effect of application of MDP containing universal adhesive was also assessed in the present study; for particular assessment of the influence of its application on the bond strength of glass ionomer restorative materials with premixed bio- ceramic MTA, and the results were compared to previously conducted studies to reach conclusive information regarding this point. Although the present study had revealed important information, yet it can be continued on by more researches to investigate the durability of the adhesive junction, as well as the effect of thermal fluctuations on the bond strength; by performing the bond strength testing after aging by thermocycling for different number cycles. Also, it is recommended to investigate the bond strength of NuSmile- Neoputty to restorative materials other than those investigated in the present study, in addition to application of different adhesive systems and strategies; in order to attain a satisfactory assessment of its bonding potential.

CONCLUSIONS

Within the limitations of the conducted study, it could be concluded that:

1- Fuji II LC glass ionomer and Cention N dental resin composite would be capable for inducing stronger bond with premixed bio- ceramic NuSmile- Neoputty material in comparison to Venus Bulk Fill dental resin composite. On the contrary, the bond strength of Equia Forte Fil to NuSmile Neoputty could not be considered satisfactory.

2- Application of MDP containing universal adhesive could be considered valuable for strengthening the adhesive junction in case of using restorative materials having resinous constituents in its composition; as Fuji II LC, Cention N, and Venus Bulk Fill. However, it would be of no value on dealing with a non resinous containing material as Equia Forte Fil.

CLINICAL RELEVANCE

On choosing among Fuji II LC or Equia Forte Fil glass ionomer; for application on top of NuSmile-Neoputty bio- ceramic material; it is recommended to use Fuji II LC, and the application of MDP containing universal adhesive would be beneficial. However, it is not advisable to use Equia Forte Fil.

Regarding dental resin composites as a restoration on top of NuSmile- Neoputty material; Cention N is preferred to Venus Bulk Fill, and application of MDP containing universal adhesive is recommended.

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