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Comparative Evaluation of Microshear Bond Strength of Adhesive Resin Incorporated with Inorganic Nanofillers Cerium Dioxide: An In-vitro Study

Dentistry Section

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ABSTRACT

Introduction: Dental adhesives are used for a wide range of clinical applications in dentistry. Bonding is required in direct composite resin restorations. In clinical situation, resin to dentin bonds are less resistant than resin to enamel bonds. Because of varying structure and composition of dentin, the failure of resin-dentin bonding resulting in staining, caries recurrence microleakage. These set of circumstances can hasten the failure and degradation of resin to dentin bonding hence inorganic fillers can be added to improve the bond strength. Cerium dioxide (CeO_2) is a rare earth oxide has been increasingly used as a nanotherapeutic material and can be successfully used in adhesive resin to improve the physical properties of adhesive resin.

Aim: To evaluate and compare microshear bond strength of adhesive resin after incorporation of inorganic nanofillers cerium dioxide.

Materials and Methods: This in-vitro study was conducted in Department of Preventive and Paediatric Dentistry at School of Dental Sciences, Krishna Institute of Medical Sciences, Karad, Maharashtra, India, from February 2022 to April 2022. The unfilled dental adhesive was used as a control (group A) and dental adhesives formulated with nanosized Cerium dioxide (CeO_2) as the inorganic filler (group B). Total 24 samples were prepared, 12 samples in each group and adhesives were evaluated for microshear bond strength. Intragroup comparison between experimental and control group was done using Unpaired t-test.

Results: The mean microshear bond strength of the adhesive system containing nanosized cerium dioxide at a concentration of 2% was 6.89±2.22 MPa (group B), which was significantly greater than that of the adhesive system without additives (group A) 4.37±1.22 MPa.

Conclusion: Microshear bond strength of the dental adhesives with nanosized CeO_2 particles, was significantly higher than the unfilled adhesive.

Keywords: Composite, Dentin bonding, Enamel bonding, Nanotherapeutic material, Nanoparticles

INTRODUCTION

Nowadays composite material and dental adhesive systems are material of choice in restorative dentistry as direct restorative materials [1]. The dentin has the wet environment which is not favourable for the polymerisation of methacrylate monomers, which promote degradation of resin dentine interface over time [2]. Along with this cavity, seal should be appropriate which is required to inactivate the residual bacteria from affected dentin and stop the further caries progression. Many modifications have been put forward in the composition of adhesive resins which improve the longevity of the restorative treatments [1]. The strategies which are currently available involves the incorporation of inorganic fillers resulting in decrease relative amount of organic phase which gives a higher hydrolytic stability to resin and a promising cavity seal [3].

Cerium dioxide (CeO_2) is found in the lanthanide series of the periodic table. It is a rare earth oxide, There are various commercial applications for CeO_2 are present. This material is most commonly used as nanotherapeutic material [4]. In dentistry, CeO_2 was used in dental ceramics since this compound resembles the natural fluorescence of the human dental enamel. Cerium has high atomic number of 58 and thus it can promote considerable attenuation of a dental X-ray beam so it is used in adhesive resin to improve the radiopacity of adhesive resin [4,5].

The heterogeneity of the structure and composition of dentin causes unfavourable environment resulting in staining, recurrent caries, Microleakage and postoperative sensitivity, and the combination of these situations can accelerate the degradation and failure of resin to dentin bonding [4]. Therefore there is need to modify the adhesive materials for breaking this vicious circle. Thus, the present study was conducted to evaluate and compare microshear bond strength of adhesive resin after incorporation of nanosized cerium dioxide particles for the first time.

MATERIALS AND METHODS

This in-vitro study was conducted in Department of Preventive and Paediatric Dentistry at School of Dental Sciences, Krishna Institute of Medical Sciences, Karad, Maharashtra, India, from February 2022 to April 2022. Ethical clearance has been obtained from the ethical committee (approval number 076/2021-2022 and reference number KIMSDU/IEC/03/2022).

Inclusion criteria: Non carious, non hypoplastic, no cracks, non restored teeth and extracted for orthodontic therapies were included in the study.

Exclusion criteria: Carious, non therapeutically extracted teeth were excluded from the study.

Total 24 samples were prepared 12 samples each group and adhesives were evaluated for microshear bond strength.

- **Group A** (n=12): The unfilled dental adhesive was used as a control group.
- **Group B** (n=12): Dental adhesives formulated with nanosized Cerium dioxide (CeO_2) as the inorganic filler.

Procedure

Preparation of Experimental Adhesive Resin: Commercialy available adhesive resin (Ivoclar vivadent Tetric N bond) [Table/Fig-1] which is 5 gms in quantity were taken into that 2wt% of nanosized cerium dioxide [Table/Fig-2] (nano research laboratory ,Maharashtra) that is 0.120 mg [4] [Table/Fig-3] of powder were mixed. In the study by Martini I et al., the cerium dioxide inorganic fillers were studied with different ranges from 0.36 to 5.76 volume% [4]. Loading higher than 2 wt% (i.e 1.44 volume %) showed decreased in the degree of conversion hence 2wt % were selected for experimental groups.

[Table/Fig-1]: Commercially available 5th generation adhesive.
[Table/Fig-2]: Nanoparticles of Cerium dioxide. (Images from left to right)

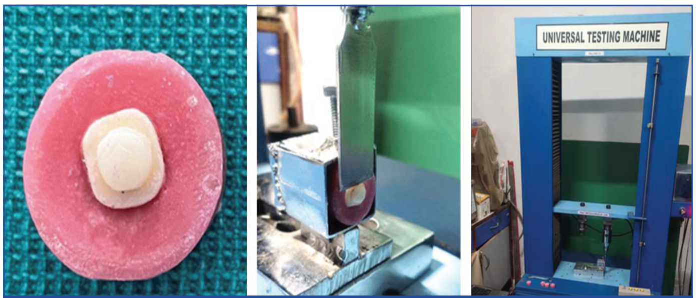
Sample preparation: Twenty four extracted premolar were included. All the debris and calculus were removed from the teeth with the ultrasonic scalers. Cleaning of teeth were done using slurry of pumice and were stored in 0.1% wt/vol thymol solution till sample preparation for parameters.

[Table/Fig-3]: Measurment of Nanoparticles of Cerium dioxide.
[Table/Fig-4]: Molar teeth showing complete removal of enamel and exposure of dentin. (Images from left to right)

Micro shear bond strength: Collected samples were taken and the enamel of the crowns were removed using a diamond disc. The tooth was then ground with 320 grit SiC paper until occlusal enamel was completely removed [Table/Fig-4].The teeth were etched with 37 wt% phosphoric acid gel for 15 s and rinsed with water, An adhesive was applied without nanofillers (group A) for 12 samples and adhesive with addition of nanosized cerium dioxide (group B) were applied on 12 samples and photo-cured for 20 seconds. A stainless steel iris with a central opening was placed on dentin surface which was treated with adhesive. The central opening of iris was filled with a composite and it was photo-cured for 30 sec [Table/Fig-5]. The samples were placed in water at 37 °C for 14 days. Then the dentin shear bond strength was measured using a chisel which was held parallel to the composite-dentin interface [Table/Fig-6] and loaded via a Universal Testing Machine [Table/Fig-7] at 0.5 mm/min until the bond failed, t is : dentine shear bond strength, $SD = 4P/(\varpi d^2)$, where d stand for diameter of the composite and P stand for load at failure [6].

STATISTICAL ANALYSIS

Statistical analysis was performed. Statistical Package for Social Science (SPSS) version 21.0 for Windows (SPSS Inc, Chicago, IL) software was used. Descriptive quantitative data was expressed in mean and standard deviation respectively. Data normality was checked. Shapiro-Wilk test were used for the same. Confidence interval is 95% and probability of alpha error (level of significance) is 5%, power of the study was 95%. Intragroup comparison between experimental and control group was done using unpaired t-test.



[Table/Fig-5]: The bonded samples with composite resin.
[Table/Fig-6]: Positioning of the sample on universal testing.
[Table/Fig-7]: Universal testing machine. (Images from left to right)

RESULTS

The results for dentin shear bond strength are plotted in [Table/Fig-8] for control group and [Table/Fig-9] for testing group. The adhesive system containing nanosized Cerium dioxide particles at a 2 wt% concentration (group B) showed a statistically significantly greater mean microshear bond strength (6.89±2.22 MPa) than that of the group A (4.37±1.22 MPa). A significant difference (p-value <0.05) between group A and group B was observed in microshear bond strength testing [Table/Fig-10].

S. No.	Sample ID	Maximum load (N)	Shear bond strength (MPa)
1	No 1	93.45	6.74
2	No 2	80.44	3.06
3	No 3	85.00	3.85
4	No 4	65.30	3.71
5	No 5	65.20	3.70
6	No 6	72.80	4.38
7	No 7	80.11	3.11
8	No 8	87.22	3.99
9	No 9	79.77	5.01
10	No 10	93.45	6.74
11	No 11	85.00	3.85
12	No 12	72.80	4.38

[Table/Fig-8]: Microshear bond strength of control group (group A).

S. No.	Sample ID	Maximum load (N)	Shear bond strength (MPa)
1	No 1	319.00	10.76
2	No 2	171.20	8.26
3	No 3	78.40	4.61
4	No 4	165.75	5.86
5	No 5	233.25	5.57
6	No 6	78.40	4.61
7	No 7	159.25	7.68
8	No 8	198.00	9.55
9	No 9	107.7	5.19
10	No 10	125.6	6.06
11	No 11	203.5	9.82
12	No 12	98.2	4.73

[Table/Fig-9]: Microshear bond strength of test group (group B).

DISCUSSION

The purpose of using dentin bonding systems which acts as an elastic intermediate layer, between the cavity walls and the

Parameters	Group A	Group B
Mean (MPa)	4.37	6.89
Standard Deviation (SD) (MPa)	1.22	2.22
Sample size (N)	12	12
Standard error of mean	0.35	0.64
Lower 95% confidence limit	3.59	5.47
Upper 95% confidence limit	5.15	8.30
Minimum	3.06	4.61
Median	3.92	5.96
Maximum	6.74	10.76
Normality test KS	0.24	0.22
Normality test p-value	0.03	

[Table/Fig-10]: Comparative values in group A and group B.

adjacent composite and between the resin and the tooth structure is to enhance the bonding strength, increase the retention of the restorative material, decrease the microleakage, dissipation of occlusal stress and prevent the polymerisation shrinkage stress [7].

In this in-vitro study, CeO_2 nanoparticles were explored. Addition of nanosized particles into the adhesive resin prevent the bond degradation by protecting the collagen fibers and improved the mechanical properties by apatite formation. This adhesive resin can also cause an increase in the pH, resulting in remineralisation of the adhesive-dentin interface and increasing the marginal and internal seals of composite restorations [8].

The micro shear bond strength test is widely used method to test the bond strength, because of the simplicity of the procedure, specimen preparation and simple test protocols [2]. In the present study, the microshear bond strength of adhesive containing nanosized cerium dioxide nanoparticles particles at a 2 wt% concentration was studied which showed the superior results than that of the adhesive system without additives [9].

Contributing factors to this effect may include the verity that number of metal-oxygen (Me-O) bonds increases because during the early stages of drying there is the release of residual water and organic solvent [10]. Hence, during the curing regimen, a further increase in cross-linking and Me-O bonding occurred because the release of water and cure time, controls the solvent from adhesive resin and thus increased the bond strength to dentin [5].

Leitune VC et al., evaluated the dental adhesive after addition of niobium pentoxide (Nb_2O_5) nanoparticles as radiopacifiers and observed that the degree of conversion decreased with the addition of particles [1]. In other study, Ta_2O_5 was added into the adhesive at weight percent of 1, 2, 5 and 10 wt %. Evaluation of radiopacity, softening in solvent, degree of conversion, and ultimate tensile strength were done. The study concluded that tantalum oxide is a promising alternative for adhesive formulation [3].

Microshear bond strength is very important once the adhesive resin is in direct contact with mineralised tissues. In this in-vitro study, incorporation of nanosized cerium dioxide particles in adhesive resin was done for the first time to best of authors' knowledge and it showed increased in bond strength of the experimental adhesive, Therefore, the experimental adhesive resins with cerium dioxide nanoparticles are suitable for further tests for degree of conversion and bioactivity.

Limitation(s)

The sample size of the study was less. Moreover, in this study, only microshear bond strength was studied. More properties of the formulation should be studied in further studies.

CONCLUSION(S)

Nanosized cerium dioxide particles were investigated for the first time as a encouraging fillers to improve the microshear bond strength of the 5th generation dental adhesives. Comparative evaluation of the microshear bond strength showed increase in the bond strength after incorporation of nanosized cerium dioxide. Cerium dioxide is a radiopaque inorganic filler with low toxicity and no enough material is available in literature, Therefore the experimental adhesive resins with cerium dioxide are suitable for further tests of degree of conversion, mineral deposition and bioactivity.

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