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Shear Bond Strength of Composite to Dentin after Various Drying Techniques and Its Micro Morphological Analysis under SEM - An In-vitro Study

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Abstract

Aim: The present in-vitro study compared the shear bond strength of composite to dentin after various methods to remove excess water from the dentin.
Materials and Methods: Sixty extracted human molars were used for the study that were randomly divided into 6 groups with 4 experimental (three-way syringe, cotton pellet, mini sponge, tissue paper) and 2 control groups (over dry, over wet). The exposed dentin surfaces were acid etched with 37% phosphoric acid for 20 seconds followed by thorough rinsing with water for 15 seconds. The teeth were kept visibly moist in all the experimental groups. Adhesive agent was applied on all specimens and restored with composite resin. The specimens were then subjected to shear bond strength tests and one specimen from each group was randomly selected for micro-morphological analysis of dentin.
Results and Conclusion: Statistical analysis was done and data were tabulated to determine average shear bond strength for each group. The results indicated that the mean values in the group using tissue paper were significantly higher than the mean values in all the other experimental groups and control groups.

Key words: Shear bond strength, Dentin, Composite

Introduction

Resin penetration into dentin with monomer impregnation of the exposed collagen resulting in formation of hybrid layer is widely accepted as an efficient method to improve resin composite bond strength to dentin. The interaction between the resin solvent and dentin surface appears to be dependent on both the individual solvent and the presence of surface moisture. Vital dentin is inherently a wet surface and a complete desiccation of dentin is difficult to achieve clinically[16].

Dehydration of the acid conditioned dentin surface through air drying is thought to induce surface tension stress, causing the exposed collagen network to collapse, shrink and form a compact coagulate that is impenetrable to resin (David.H.Pashley, 1993)[11]. If some water remains inside the interfibrillar spaces, the loose quality of the collagen matrix is maintained and the interfibrillar spaces are left open. An appropriate amount of moisture on the dentin surface has been reported to promote polymerization reaction of specific monomers. Hence it should be emphasized that, this wet-bonding technique can guarantee efficient resin interdiffusion only if, all of the remaining water on the dentin surface is eliminated as completely as possible and replaced by monomers during the subsequent priming step[16].

Clinically, a shiny hydrated surface is seen with moist dentin. However, the amount of water present on the dentin surface may be a negative factor and there is a limit to the amount of moisture necessary for keeping the demineralized collagen stable[12]. Various procedures used to remove the excess water from the dentin surfaces are: drying the dentin surface with compressed air, wiping the dentin with damp cotton pellet or blotting the surface with a tissue paper[5].

Hence the purpose of this in-vitro study was to compare the shear bond strength of resin composite to dentin substrate after various drying techniques keeping the substrate visibly moist and its micro morphological analysis under scanning electron microscope.

MATERIALS AND METHODS

Methodology

A total of 60 extracted human molars were used for the study. The teeth were thoroughly debrided using ultrasonic scaler tips and were stored in distilled water, until ready for use. Each tooth was then mounted in an auto polymerizing acrylic resin, using wax blocks (Fig 1A and 1B).The occlusal surfaces of the teeth were ground at slow speed using a diamond disc under running water to create flat dentin surface. The exposed dentin surfaces were then acid etched using 37% phosphoric acid for 20 seconds followed by thorough rinsing with water for 15 seconds. The teeth were randomly assigned into 6 groups of 10 teeth each. The conditioned dentin surfaces were then kept visibly moist in all experimental groups and excess water removal was done using various drying techniques.

In **Group-I** the etched dentin surfaces were gently blow dried to remove the excess water for three seconds using oil free compressed air. A layer of bonding agent (single Bond) was applied as per the manufacturer's instructions with a fully saturated brush tip and was left undisturbed for 20 seconds .Further a second coat was applied, gently blow dried for 2 seconds to remove excess solvent and light cured for a period of 20 seconds (Fig 1C)

A metallic ring measuring 2mm height with an internal diameter of 3mm and thickness of 5mm was placed on the conditioned dentin surface and resin composite (3M ESPE) was packed into the ring and polymerized for 40seconds using a light curing unit (3M ESPE, 2500 Curing light). In **Group-II**, the excess water from the etched dentin surfaces were removed using a hydrophilic cotton pellet by gently applying pressure on the dentin, only once. Rest of the procedures were carried out as in Group-I (Fig 1D). In **Group-III,** the excess water from the etched dentin surfaces were removed using a minisponge by gently applying pressure on the dentin using a tweezer. Rest of the procedures were carried out as in Group-I (Fig 1E). In **Group-IV**, the excess water from the etched dentin surfaces were blot dried using a tissue paper by gently applying pressure on the dentin. Rest of the procedures was carried out as in Group-I (Fig 1F). In **Group V** and **Group VI** (control groups), the etched dentin surfaces were over dried using a three-way syringe with oil-free compressed air for a period of 5 seconds from a distance of 3 cms and the etched dentin surfaces were left without removing the excess water. Rest of the procedures were carried out as in **Group-I**.

Testing procedures

Each specimen was loaded in a universal testing machine (Lloyd universal testing machine). The bond strength was measured in the shear mode at a cross head speed of 1.0mm/min.

Evaluation of shear bond strength

The force required to dislodge the composite cylinder from the conditioned dentin surfaces were recorded in Newtons and shear-bond strength in MPa which was calculated from the following equation:-

Value obtained in Kg * 9.81 = Newton (Force)

Bond Strength (MPa) = Maximum load (Newton's)/ Surface area of the Cylinder.

Specimen preparation for scanning electron microscopy

One specimen from each group was randomly selected for the micro morphological analysis. The teeth were sectioned longitudinally through the restorations in a bucco-lingual plane with a diamond disc under running water. The samples were then polished with a wet #600 grit silicon carbide paper. After thorough rinsing, the samples were demineralized for 30 seconds with 6N hydrochloric acid, rinsed again, deproteinized with 2.5% NaOCl for 10 minutes, and then left to air dry in a desiccator under low vacuum pressure for 24 hours. Following the drying procedure, the samples were sputter-coated with gold and viewed under SEM.

RESULTS

The results obtained were tabulated (Table 1&2); graphically represented and statistical analysis was done to determine the average shear bond strength for each group.

(Table-1) Values in (MPa)

Sl. No.	Group I	Group II	Group III	Group IV	Group V	Group VI
1	11.07	14.12	14.63	16.98	10.16	11.28
2	11.26	14.84	13.92	17.12	11.28	10.98
3	12.01	13.97	14.24	16.86	10.86	11.48
4	11.86	14.82	14.84	16.98	10.12	11.52
5	11.08	14.63	14.58	16.86	10.89	11.26
6	12.15	13.94	14.69	17.23	11.28	10.94
7	11.36	14.82	13.98	17.89	10.64	11.24
8	11.03	13.18	13.64	17.12	11.38	10.78
9	11.12	13.98	14.84	16.96	11.02	11.84
10	11.82	14.64	14.28	17.82	10.98	11.28

Statistical analysis

Mean and standard deviation were estimated from the sample for each study group. Mean values were compared between different study groups by one-way ANOVA followed by Tukey-HSD procedure.In the present study, $P<0.05$ was considered as the level of significance. Multiple range test by Tukey-HSD procedure was employed to identify the significant group at 5% level.

(Table-2) Mean, standard deviation and test of significance of mean values among different study groups.

Group	Mean ± SD	P – value	Significant groups at 5% level
I	11.48±0.44		IV Vs I, II, III, V, VI
II	14.29±0.55		III Vs I, V, VI
III	14.36±0.42		II Vs I, V, VI
IV	17.18±0.37		I Vs V
V	10.86±0.44		
VI	11.26±0.31	<0.0001	

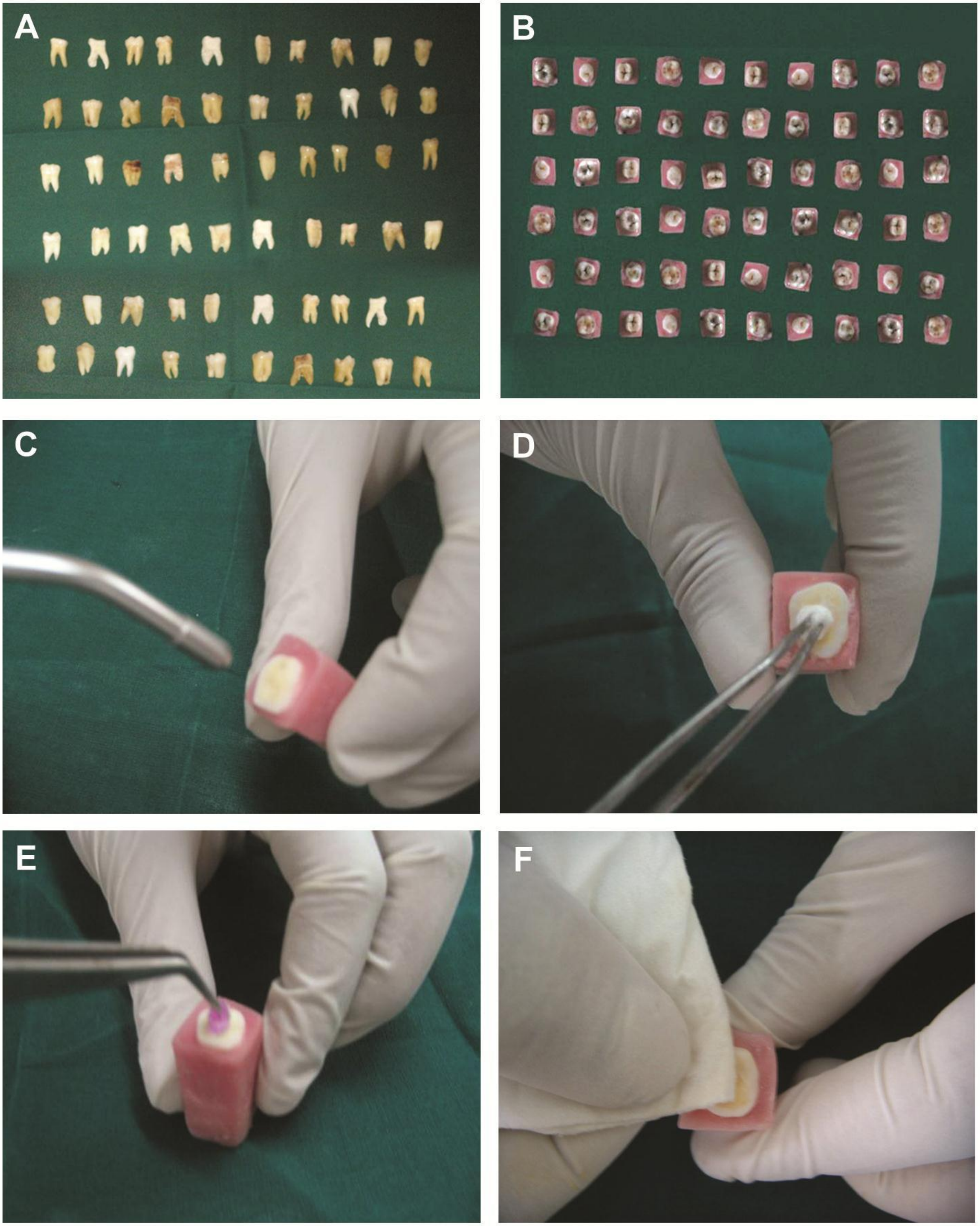


Fig 1A and 1B: Sixty extracted human molars selected that were used for the study, randomly divided into 6 groups (4 experimental and 2 control groups).

Fig 1C to 1F: Demonstration of drying techniques used in Group I to Group IV study.

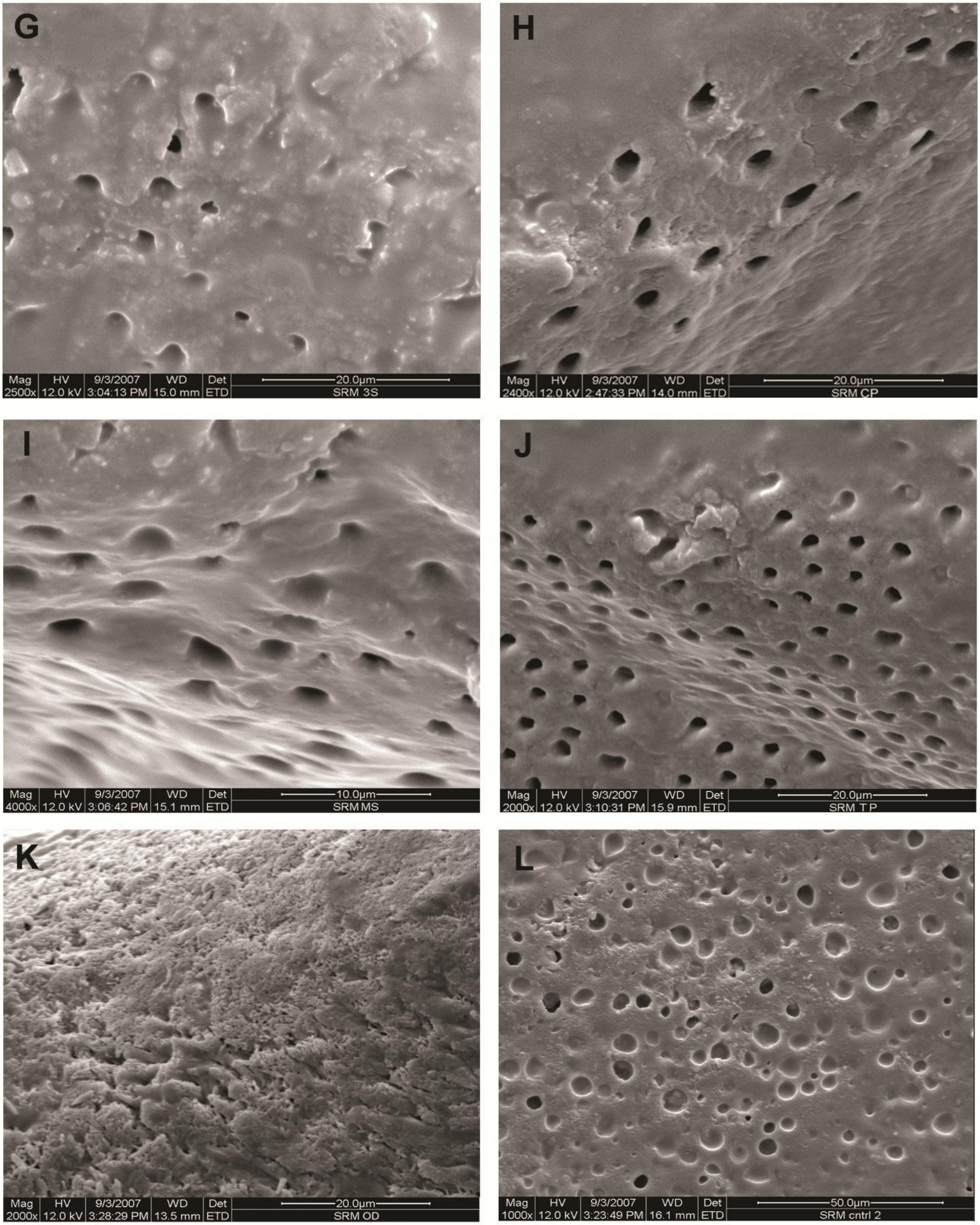
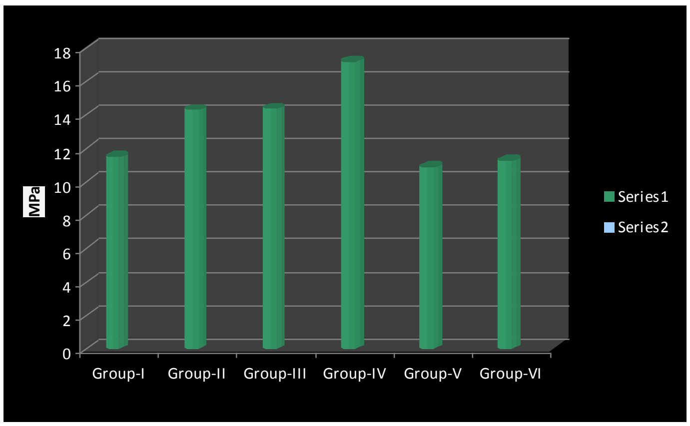


Fig 1G to 1L: SEM Results showing difference in the characteristics of the dentinal tubules to various drying techniques.

Inference

Mean values in Group IV (tissue paper) (17.18 ± 0.37) was significantly higher than the mean values in Group I (three-way syringe) (11.48 ± 0.44), Group II (cotton pellet) (14.29 ± 0.55), Group III (mini sponge) (14.36 ± 0.42) and the control groups, Group V (over dry) (10.86 ± 0.44), and Group VI (over wet) (11.26 ± 0.31) ($P<0.05$).

The mean values in Group II (cotton pellet) and similarly in Group III (mini sponge) were significantly higher than the mean values in Group I (three-way syringe), and control groups Group V (over dry), and Group VI (over wet) ($P<0.05$). Further, the mean value in Group I (three-way syringe) was significantly higher than the mean value in the control group (over dry) ($P<0.05$). However, there was no significant difference in mean values between any other groups ($P>0.05$).



Graph- Mean rank of scores of all experimental and Control groups

SEM OBSERVATIONS OF BONDED SPECIMENS

Fig 1G shows the SEM view of a specimen bonded under blow dried (3 seconds) condition using a three-way syringe. Few dentinal tubules that are partially open due to collapsed dentin matrix and absence of bonded collagen and interfibrillar spaces are seen. Fig 1H shows the SEM view of bonded dentin substrate after removing the excess water using a damp cotton pellet (Group-II). The number of open dentinal tubules was slightly more when compared to Group-I (three-way syringe). Fig 1I shows the SEM view of bonded dentin substrate after removing the excess water using a mini sponge (Group-III). The number of open dentinal tubules was similar as seen in Group-II (cotton pellet), but more when compared to Group-I (Three-way syringe). Fig 1J shows the SEM view of a bonded dentin substrate after removing the excess water using a tissue paper (Group-IV). The number of open tubules was more when compared to Group I, II& III. Resin tag penetration is also seen. Fig 1K shows the dentin surface of over dried (Group-V) specimen. Occluded dentinal tubules due to collapse of collagen fibrils forming a compact coagulate that is impenetrable to resin is evident. Fig 1L shows the dentin surface of over wet (Group-VI) specimen showing blister/globule formations.

DISCUSSION

The mechanism of dentin bonding to most adhesive systems is based on hybridization. Hybridization of dentin is a process that creates a molecular level mixture of adhesive polymers and dental hard tissues. The relationship between bond strengths and hybrid layer formation differ depending on the moisture content of the dentin. Hence, maintaining a moist surface for a hydrophilic bonding system is necessary for optimal development of the intermingled interdiffusion zone or hybrid layer (Van Meerbeek, Nakabayashi 1992, 1982). This clinical technique commonly referred to as "wet bonding" was introduced by Kanca and Gwinnett in the early 1990's[16].

This wet-bonding technique has been shown repeatedly to enhance bond strengths because water preserves the porosity of collagen network available for monomer interdiffusion[7, 8]. An appropriate amount of moisture on the dentin surface has also been reported to promote polymerization reaction of specific monomers[16].

In the present in-vitro study, molars were selected for the bond strength tests because it provided a greater surface area after the dentin surfaces were flattened which enabled the composite posts to be bonded over the adhesive. Additionally the combination of bond strength data with micro morphological analysis of adhesive interfaces provides useful information concerning the interaction of dentin bonding systems with dental substrates[15].

In this study fifth-generation adhesive system was used because studies revealed that it produced superior bond strengths (14-25MPa) when compared to self etching adhesives[9]. Fifth generation adhesive also contains ethanol which acts as a water chaser. Because of this, it is less sensitive to moisture conditions, and thus facilitates adequate wettability.

The present study compared the shear bond strength of resin composite to dentin using various drying techniques to remove excess water from the dentin surface while keeping the dentin visibly moist. The results of the study showed that, blow drying for three seconds using oil-free compressed air (Group-I) showed the lowest shear bond strength of resin composite to dentin when compared to all the other experimental groups (Group-II, Group-III, Group-IV). This might be due to the absence of bonded collagen and interfibrillar spaces within the treated dentin suggesting the existence of collapsed dentin matrix. This restricts resin permeation into the surface intertubular matrix producing an incompletely infiltrated area called hybridoid region (Tay FR, Gwinett 1996)[6,16]. In Group-IV the excess water blot dried using tissue paper (Group-IV) showed the highest bond strength when compared to all the other groups, because as discussed earlier the interfibrillar spaces within the collagen network was maintained from collapsing during primer application[16].

However, there was no significant difference in bond strength when pooled moisture was removed using cotton pellet (Group-II) and mini sponge (Group-III). In the present study, control groups which included over dried (Group-V) and over-wet (Group-VI) specimens showed least shear bond strengths when compared to the experimental groups. The possible explanation is that, over drying or desiccation of the dentin is thought to induce surface tension stress, causing the exposed collagen network to collapse; shrink and form a compact coagulate that is impenetrable to resin (Linlin 2000)[13]. Whereas over wetting of the dentin appears to cause phase separation of the hydrophobic and hydrophilic monomer components, resulting in blister and globule formation spaces at the resin dentin interface (Tay and other 1996)[16,12]. Such interfaces cause incompletely sealed tubules thereby weakening the resin dentin bond.

The present bond strength study tends to confirm with the SEM studies, as well as other bond strength studies that, with a moist surface, optimum infiltration of adhesive resin into the demineralized layer occurs and higher bond strength values were achieved (Kanca in 1996, Swift & Triolo in 1992)[5,8,12].

CONCLUSION

Within the limitations of the present in-vitro study, it is concluded that blot drying with tissue paper produced the highest shear bond strength of single bond adhesive to dentin while a 3 second gentle air drying at a distance of 3 centimeters from the substrate produced the weakest bond strength. There was no significant difference in shear bond strength between Group II (cotton pellet) and Group III (mini sponge) after excess water removal.

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