



# **Comparative Evaluation of Surface Roughness Using Two Different Mechanical Surface Treatments and their Effect on Microshear Bond Strength Using Different Nanocomposites: An *In vitro* Sem Study**

**Prabhavathi Poornima<sup>a++</sup>, B.S. Keshava Prasad<sup>b#</sup>  
and Kuchimanchi Sneha Aishwarya<sup>b†\*</sup>**

<sup>a</sup> DAPM RV Dental College Bangalore, India.

<sup>b</sup> Department of Conservative Dentistry and Endodontics, DAPM RV Dental College Bangalore. India.

## **Authors' contributions**

*This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.*

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<sup>++</sup> MDS and Endodontist;

<sup>#</sup> MDS, Professor and Head of the Department;

<sup>†</sup> Post Graduate and MDS;

\*Corresponding author: Email: [kuchimanchisnehaaishwarya@gmail.com](mailto:kuchimanchisnehaaishwarya@gmail.com);

## ABSTRACT

**Introduction:** This study compared the impact of surface roughness on the microshear bond strength of nanocomposites to dentin that underwent airborne abrasion and conventional rotary tools as two distinct mechanical surface treatments. The study also sought to determine how well the Self Etch Adhesive method worked in creating a strong bond following dentinal air abrasion.

**Materials and Methodology:** Forty six extracted human molars were used for this in-vitro study. The occlusal third of the crown was sectioned to expose the dentin and the specimen were randomly assigned into two main groups based on the mechanical surface treatment. Each group was further subdivided into two subgroups based on the nature of composite resin used. Group 1A : abrasion with diamond bur followed by restoration with nanohybrid resin. Group 1B: abrasion with diamond bur followed by restoration with nanofilled resin. Group 2A: air-borne dentin abrasion followed by restoration with nanohybrid resin. Group 2B: air-borne dentin abrasion followed by restoration with nanofilled resin.

**Results:** Results showed that Group 1A demonstrated significantly higher mean Microshear bond strength as compared to Group 2A & Group 2B at  $P < 0.001$ . This was followed next by Group 1B showing significantly higher mean Microshear bond strength as compared to Group 2A & Group 2B at  $P < 0.001$ . These results infer that air abrasion with aluminum oxide particles decreased the bond strength for the self adhesive system tested, irrespective of the type of nanocomposite used when tested under UTM. The study also concluded that the Nanohybrid composites performed better in terms of bond strengths in comparison with the Nanofilled composites regardless of the surface treatment.

*Keywords: Aesthetic restorative materials; microshear bond; nanocomposites.*

## 1. INTRODUCTION

“With the advancement of aesthetic restorative materials, adhesive systems have become essential elements in various clinical applications. Adhesive systems are responsible for the bonding of restorative material to dental structures. Dental adhesive technology has evolved towards complex formulations with simplified clinical procedures” [1] “In the prosthetic and restorative dentistry fields, the use of adhesive materials has made it possible to increase the retention of the restorations, improve the aesthetic results, as well as reduce the invasiveness. Till date, the use of adhesive materials have led to good outcomes both in terms of mechanical properties and adhesive capabilities” [1,2].

“Dentin adhesion occurs by formation of a hybrid layer / hybridization - superficial demineralization followed by infiltration of resin monomers into the dentinal tubules that upon setting, form resin tags that micromechanically get interlocked into the dentin” [3].

“However, various factors can influence the long-term performance of adhesive restorations, such as the vulnerability of the adhesion interface to oral fluids, the adhesive technique used, the material and the quality of the restoration, and

the treatment of the residual dental tissue. In case of failure, the main effects involve the resin-dentin bonding and detachment of the restoration, as well as recurrent caries. The sum of these factors can reduce the survival rate of the bonded restorations” [4].

“Smear layer, a structure formed by the debris resultant from the cutting process, may present different composition, thickness and morphology, depending on the location and type of burs used” [5]. “As described in previous studies smear layer can act as a physical barrier and obliterate the dentinal tubules entrance, reducing the permeability of the tubules to the penetration of the adhesive system and therefore making the adhesion to dentin substrate dependent on the type of smear layer pretreatment. Therefore, dentin surface treatments for smear layer cleaning, such as its complete removal, dissolution, replacement or modification, should be considered as a decisive step previous to restoration bonding procedures” [5,6]

“The main strategies to achieve effective dentin adhesion are: etch and-rinse protocol which requires the conditioning of substrate with phosphoric acid, thus removing the smear layer; and self-etch mode which uses a non-rinse acidic primer, leading to a smear layer dissolution, with

no demineralization of the subsurface and the promotion of resin infiltration. Acid etching of dentin, which removes the smear layer completely and demineralizes the subsurface, is an established and predictable clinical procedure, but features inherent to dentin conditioning can influence the bonding performance of adhesives. Dentinal collagen exposed by an etch-and-rinse adhesive has been found to be highly vulnerable to hydrolytic and enzymatic degradation processes” [7].

“A promising approach to adhesion is the use of one-step self-etch adhesives that slightly demineralize the dentin surface and simultaneously provide resin infiltration. When using self-etch adhesives, a hybrid layer is formed with the smear layer incorporated. Self-etch adhesives can improve dentin bonding strength and provide adhesion to dentin comparable or even superior to bonds obtained with adhesive systems that advise acid-etching as a separate step of the bonding protocol” [8].

“Over the years, different adhesion protocols, alternative materials, and substrate pretreatment procedures able to influence the degree of adhesion have been studied. In this regard, chemical or mechanical dentin pretreatments are procedures that increase the roughness of the treated surface and may influence the adhesion strength of a bonding agent by increasing the contact area between the dentine and the adhesive surface. Besides the requirement for micromechanical adhesion, pretreatments can be used with the aim of removing debris that can impair the final bonding restoration” [9].

Among the various pre-treatments, the most commonly used methods to roughen the surface over the years in clinical scenarios is use of rotary instruments that forms macro-retentive surfaces. Although Air abrasion was first described by Black in the 1950s, it was put aside due to the introduction of various high-speed rotary cutting bur. However, Air abrasion technique has recently re-emerged after modifications as a special hand piece at the chair side with aluminium oxide particles, being propelled by high velocity air pressure that provides ultrafine mechanical retention that may be more conducive to bonding.

“This alternative technology improves the patients’ comfort by reducing pressure, heat,

vibration, and noise. In addition to the possibility of increasing adhesion of restorative materials to tissues, air abrasion is also indicated for the removal of caries and restorative materials, repair of ceramic restorations and surface treatment of enamel and dentin” [10,11].

“Although a wide range of resin based materials are available, several types of composite materials are manufactured that greatly differ among each other in terms of characteristics of their inorganic fillers, which influences the viscosity, physical properties hence effecting the clinical performance of the restoration” [12].

“The recent evolution of fillers have turned to the production of nano particles, that are incorporated into the composites leading nanofilled composites containing nanosized fillers throughout the resin matrix and the nanohybrid composites that combine nanomeric and conventional fillers similar to the microhybrid composites” [13].

As there is inadequate literature with regard to the use of nanocomposites and self etchant after dentinal surface air abrasion, an attempt is made to study the bond strength of two new nanocomposites using self-etchant, after mechanical surface treatments with air abrasion and conventional method. The objective of the study is:

1. To compare and analyze the surface roughness produced on the dentin by two different mechanical surface treatments using the conventional rotary instruments and air-borne abrasion under SEM.
2. To compare and analyze the effect of surface roughness on the microshear bond strength of nanofilled and nanohybrid composite resin using self etchant adhesives on dentin.

## 2. MATERIALS AND METHODOLOGY

1. Forty six human molars that are extracted for periodontal reasons were collected and stored in distilled water until use. Teeth without fractures or cracks, without caries or restoration and without developmental anomaly were included in the study.

**Specimen selection and preparation:** Forty six Permanent molars were extracted and stored in distilled water. The teeth first were cleared of adherent patient material by scrubbing with detergent and water followed by cleansing with

an ultrasonic scaler. Then they were immersed in 10% formalin for a week. The occlusal third of the crown were sectioned using a double sided diamond disk at low speed and cooled with water continuously. The exposed dentinal surface were polished with #600 grit SiC paper in circular motion for 60 seconds, under running water to obtain a standardized smear layer.

**Experimental protocol:** Forty six specimens were randomly assigned into two main groups based on the mechanical surface treatment. Each group were further subdivided into two subgroups based on the nature of composite resin used.

Group 1A: Dentin abraded with diamond bur followed by application of adhesive and restoration with Nanohybrid resin

Group 1B: Dentin abraded with diamond bur followed by application of adhesive and restoration with Nanofilled resin

Group 2A: Dentin abraded with Aluminium oxide air-borne abrasion followed by application of adhesive and restoration with Nanohybrid resin

Group 2B: Dentin abraded with Aluminium oxide air-borne abrasion followed by application of adhesive and restoration with Nanofilled resin

**Dentin pre-treatment:** Group 1: A fine grit (53-63micron) diamond bur was mounted in a high speed turbine. For standardization, dentinal surface of each specimen was abraded by passing the bur on the dentin for 10 seconds in the same direction as uniformly as possible, by using light pressure and keeping the active parts of the bur always in contact with the dentinal surface.

Group 2: The tip of air abrasion hand piece was positioned 5mm from the dentin surface, at 90 degree angulation. A gutta percha cone measuring 5mm was used to standardize this distance. Abrasion was performed with Aluminium oxide particles of 27.5 micron, under 5 bar air pressure for 10 seconds.

After the above surface treatments, the dentin surface was gently rinsed and blot dried. A coat of adhesive was applied with an applicator and was left undisturbed for 10 seconds, after which the surface was strongly air dried

for 5 seconds and light cured for 20 seconds.

cured for 20 seconds. These specimens were stored in distilled water at 37degree Celsius for 24 hours. The specimens were cross-sectioned perpendicularly to the adhesive-tooth interface, to produce 46 dentin-composite resin blocks with a sectional square area of approximately 25 square mm and length of the block being approximately 8mm. 10 blocks were assigned to each sub group. Then the roots were separated from the crown above the cementoenamel junction releasing the dentin-composite blocks. The blocks were then examined under stereomicroscope in order to check for defects, cracks and to eliminate such specimens if any.

Three specimens from each main group were kept aside for evaluation of surface roughness under SEM, the rest forty specimens were subjected to testing of microshear bond strength. The specimens were subjected to thermocycling aging. Water storage at 37°C for 1 day were performed prior to thermal cycling. The specimens were immersed alternately in water baths of 5 and 55°C, for a total of 500-1000 cycles, using a sieve for storage and transportation. The cycle duration was 1min with a dwell time in each water bath of 28 seconds.

Each specimen were embedded in acrylic resin block. Holes were drilled in the resin to allow stainless steel wire to stabilize the sample and transfer the shear load to the sample. The wires from both the ends of the block were fixed on to the holder of the device. Specimens were suffered in a shear mode in a universal testing machine (Instiron Multitest 10) at a 1mm/ min speed for fracture assessment. After Microshear bond strength testing, the fractured blocks were examined under SEM magnification and the failure mode was identified.

Failure types were classified as follows:

- a. Adhesive (fracture at the interface between resin and dentin)
- b. Cohesive (fracture within the body of dentin or resin)
- c. Mixed (adhesive fracture combined with cohesive fracture)

**Scanning electron microscopy:** Three samples from both the main groups were subjected to SEM (JCM 6000 PLUS) by mounting on a specimen stub and were sputter-coated with gold-palladium before SEM analysis.



**Fig. 1. Selected sample teeth**



**Fig. 2. Sample decoronation at occlusal third**



**Fig. 3. Dentin pretreatment with diamond point**



**Fig. 4. Chair side air abrasion unit**



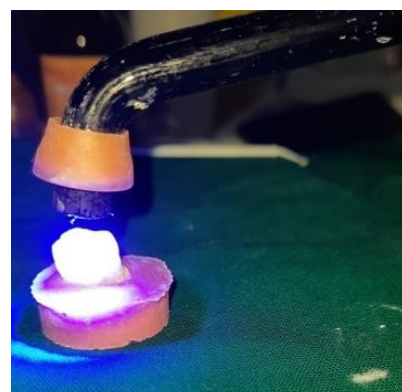
**Fig. 5. Dentin pretreatment with aluminium oxide air abrasion**



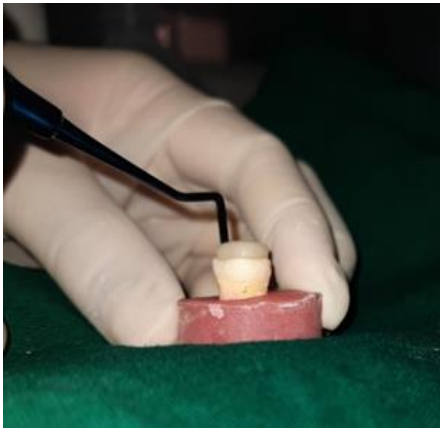
**Fig. 6. Materials used for adhesive application and composite build up**



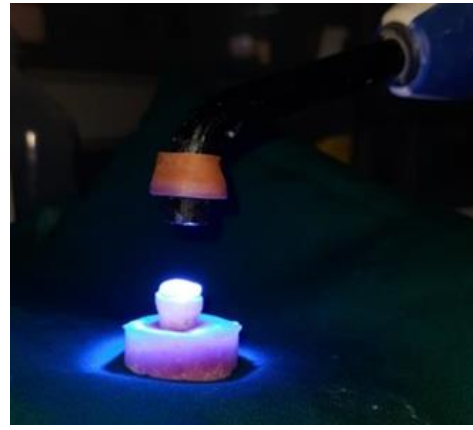
**Fig. 7. Application of Self Etch Adhesive**



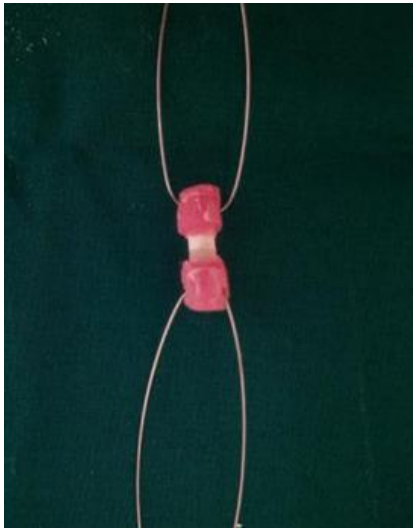
**Fig. 8. Light curing for 20sec**



**Fig. 9. Incremental composite build up**



**Fig. 10. Light curing for 30 seconds**



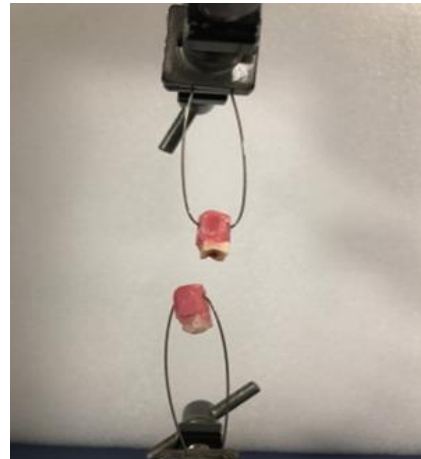
**Fig. 11. Sample mounted to acrylic and engaged with wires**



**Fig. 12. Universal testing machine**



**Fig. 13. Sample subjected to Microshear stress**



**Fig. 14. Fracture of specimen**



**Fig. 15. Scanning electronic microscope**

### 3. RESULTS

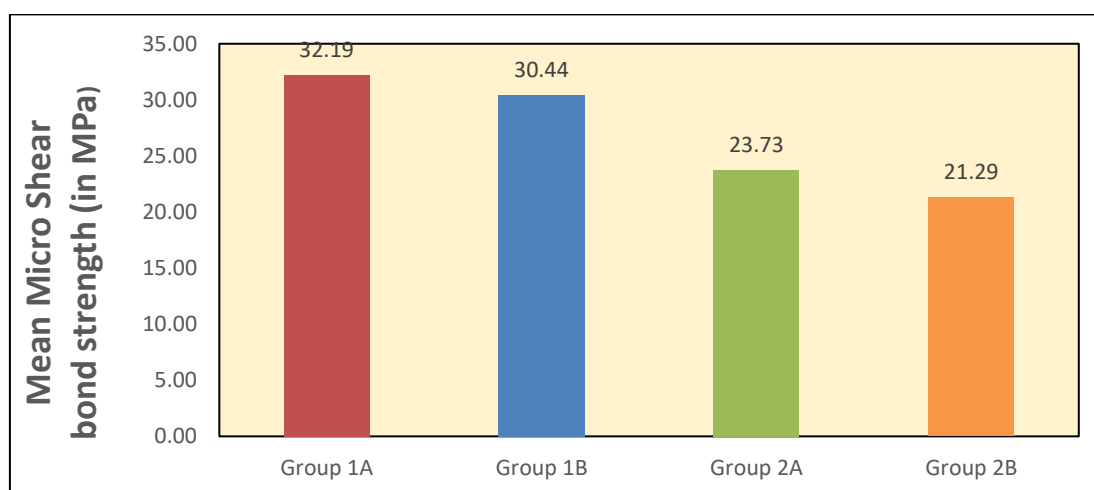
One-way ANOVA test followed by Tukey's post hoc test was used to compare the mean Micro shear bond strength (in MPa) between different groups. Chi Square test was used to compare the modes of failure between 4 study groups. The level of significance was set at  $P < 0.05$ .

The study compared and analyzed the surface roughness produced on the dentin by using the rotary instrument and air-borne abrasion under SEM. The study also analyzed the effect of surface roughness on the microshear bond strength of nanofilled and nanohybrid composite resin using self etchant adhesive on dentin.

The test results demonstrate that the mean Microshear bond strength for Group 1A was

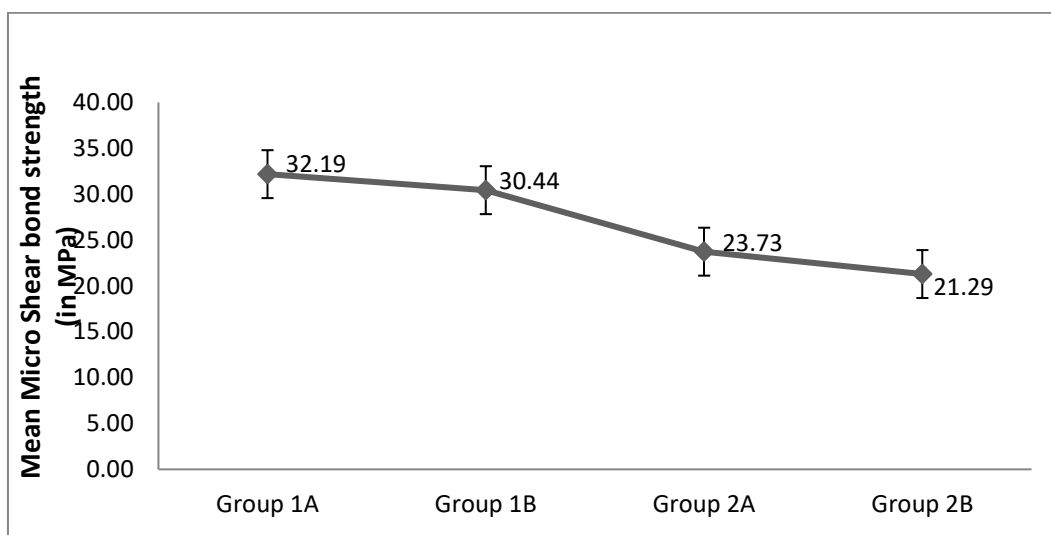
$32.19 \pm 2.61$ , Group 1B was  $30.44 \pm 2.33$ , Group 2A was  $23.73 \pm 1.20$  and in Group 2B was  $21.29 \pm 1.49$ . This difference in mean Microshear Bond Strength between 4 groups was statistically significant at  $P < 0.001$ , as shown in and Graph no.1.

Finally, Group 2A also showed significantly higher mean micro tensile bond strength as compared to Group 2B at  $P = 0.04$ . Group 1A showed relatively higher mean Microshear bond strength as compared to Group 1B. However, no significant difference was observed between Group 1A and Group 1B [ $P = 0.22$ ]. The results infer that Group 1A showed significantly higher mean Microshear bond strength, followed by Group 1B, Group 2A & least with 2B., as shown in Graph. 2.

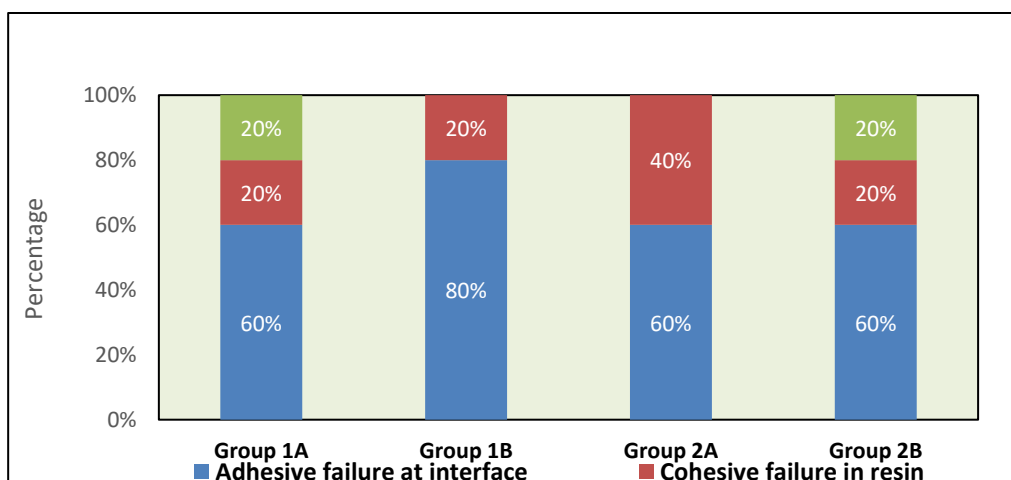


**Graph. 1. Mean microshear bond strength (in MPa) between different study groups**

Note \* - Statistically Significant Group 1A – Bur + Nanohybrid; Group 1B – Bur + Nanofilled; Group 2A – Air abrasion + Nanohybrid and Group 2B – Air Abrasion + Nanofilled

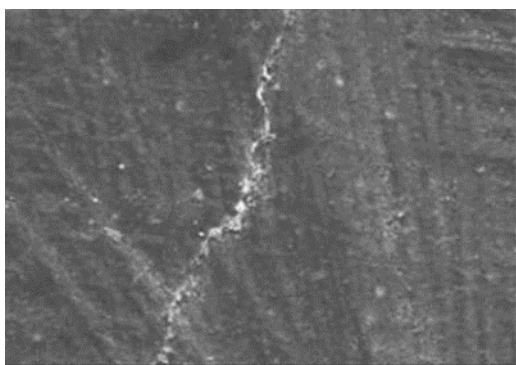


**Graph. 2. Micro Shear bond strength (in MPa) between different study groups, with standard deviations**

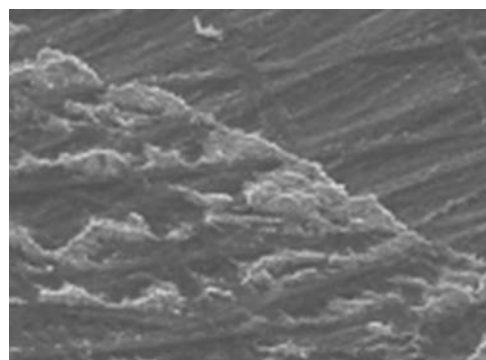


**Graph. 3. Mode of Failure between different study groups**

Scanning electron microscope images showing modes of failures:

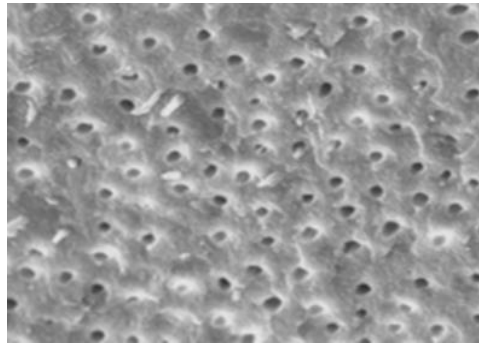


**Fig. 16. Cohesive failure in the resin**



**Fig. 17. Adhesive failure at interface**





**Fig. 18. Cohesive failure in dentin**

Comparison of mode of failure: The test results showed that in all the groups, Adhesive failure at interface was more predominant as compared to cohesive failure, ranging between 60 – 80%. Group 2A demonstrated relatively increased percentage of cohesive failure in resin [40%] as compared to other groups [20%]. Cohesive failure in dentine was only seen in Group 1A & Group 2B with 20% each, as shown in Graph. 3.

#### 4. DISCUSSION

“Various factors can influence the long-term performance of adhesive restorations, such as the vulnerability of the adhesion interface to oral fluids, the type of adhesive and technique used, the material and the quality of the restoration, the treatment of the residual dental tissue. In case of failure, the main effects involve the resin–dentin bonding and detachment of the restoration, leading to recurrent caries. The sum of these factors can reduce the survival rate of the bonded restorations” [7].

“Due to the nature of dentin substrate, namely its morphologic characteristics, higher organic content, the presence of fluid in dentinal tubules and presence of smear-layer, bonding to dentin still lacks improvements that guarantee the durability of the adhesive interface. Smear layer, a structure formed by the debris resultant from the cutting process, may present different composition, thickness and morphology, depending on the location and type of burs used. This can obliterate the dentinal tubules, reducing their permeability to the penetration of the adhesive system. Therefore adhesion to dentin substrate also depends on the type of smear layer pretreatment” [3,4].

Self-etch systems incorporate a non-rinse acidic primer that dissolves the smear layer with minimum subsurface demineralization and

simultaneous promotion of resin infiltration. Hence the hybrid layer formed incorporates the smear layer. The primers are formed by aqueous mixtures of acidic functional monomers, generally phosphoric acid- or carboxylic acid-esters, such as 10 -Methacryloyloxydecyl Dihydrogen Phosphate (10-MDP), create ionic bond between their functional groups and hydroxyapatite calcium.

“Co-solvents, such as acetone or ethanol, are frequently added to form an azeotropic solution with water, thus promoting solvation of hydrophobic components and ensuring proper dentin wettability. Hence, the level of dentin moistness is no longer a critical issue to the bonding procedure reducing the sensitivity” [12,13,14,15].

Results of the present study showed that Group 1A (Bur + Nanohybrid) demonstrated significantly higher mean Microshear bond strength as compared to Group 2A (Air abrasion + Nanohybrid) & Group 2B (Air Abrasion + Nanofilled) at  $P < 0.001$ . This was followed next by Group 1B (Bur + Nanofilled) showing significantly higher mean Microshear bond strength as compared to Group 2A & Group 2B at  $P < 0.001$ . These results could infer that air abrasion with aluminum oxide particles as a mechanical pretreatment method decreased the bond strength for the self adhesive system tested, irrespective of the type of nanocomposite used when tested under UTM.

SEM images of the resin dentin interface revealed that the self-etch adhesive was apparently unable to dissolve the dense and amorphous smear-layer produced by  $Al_2O_3$  abrasion, hampering the resin monomers infiltration. On the contrary, where dentin was not air abraded, the adhesive interface was well-defined, the hybrid layer being more

homogeneous and continuous, presenting with more number of resin tags and opened dentinal tubules with funnel shape, ascribed to an adequate adhesive pattern.

“This was in accordance with a previous study done by Cruz et al., where they described that the creation of dense and amorphous smear layer after air abrasion, altered the pattern of interaction between the adhesive systems and the substrate which in-turn negatively influenced the adhesion” [16].

In a study, Sutil et al. stated that, “when the ScotchBond™ Universal adhesive was used in the etch-and-rinse mode,  $\mu$ -TBS increased significantly when the dentin was abraded with  $Al_2O_3$ ”. “However, when ScotchBond™ Universal was used in self-etch mode, air abrasion did not increase dentin bond strength. The roughened surface created by air abrasion restricted the penetration of the adhesive monomer when the modified dentin surface was not etched with phosphoric acid, compromising the adhesive layer durability” [17-19]. The increase in bond strength observed for the SbU in etch-and-rinse strategy may be due to a change in the dentinal surface energy caused by abrasion with aluminum oxide that promoted better interactions between forces of cohesion and adhesion which determine whether wetting (the spreading of a liquid over a surface) occurs, and increasing area available for adhesion” [20].

“In addition, acid conditioning removes the smear layer and can remove aluminum oxide particles left on the dentinal surface, thus exposing the dentinal tubules, improving the infiltration of the adhesive into the dentin and enhancing resin tag formation” [21-23]. Similar findings were obtained in the present study where air abrasion followed by application of Self Etch Adhesive seemed to have deleterious effects on adhesion to dentin.

Self-etching systems have the advantage of lesser chair side application time with minimal technique sensitivity in comparison to etch and rinse systems. Eliminating the acid etching step decreases the risk of collagen network collapse, since the carboxylate or phosphate acidic groups simultaneously etch and prime the dentin substrate [11].

The bonding mechanism of self-etch adhesive systems has been intensely investigated and two-fold bonding mechanisms; micro-mechanical

interlocking and chemical bonding were described, which seems to be advantageous in terms of restoration durability. The micro-mechanical bonding contributes to provide strength against mechanical stress, while the chemical interaction reduces hydrolytic degradation, keeping the marginal sealing of restorations for a longer period [19].

However, some failures of restoration using this type of adhesive can be associated with the inability of the adhesive to penetrate correctly into the smear layer and reach the underlying dentin. In addition, it might be difficult to completely evaporate the water during the air-drying step, leading to an incomplete adhesive polymerization and increased hydrolysis, thus compromising the bonding durability [24].

The air abrasion has been widely used in the recent years, in combination with biomimetic restorations as a conservative approach to generate a roughened dentin surface, increasing the contact area for adhesion and thus improving the interfacial contact between substrate and adhesive. According to Rafael et al.,  $Al_2O_3$  air abrasion as a dentin pre-treatment allows the preservation of intertubular dentin, maintaining the original diameter of dentinal tubules entrances [25].

Particle size may not directly influence adhesion strength however, several studies have stated that smaller particles create a more retentive pattern and may lead to a stronger adhesive interface. Hence smaller particle size was chosen for this study. As the particles reach the dentin, the kinetic energy is released, resulting in microscopic fractures of the surface. This is hypothesized to allow an enhancement of mechanical interlocking between resin monomers and intertubular dentin, promoting higher bond strength values [26].

As a disadvantage, it is thought that the permanence of the  $Al_2O_3$  debris on the dentin surface may influence the penetration of resin monomers, presenting a risk for the adhesion success. Some authors advise to perform dentin etching after  $Al_2O_3$  air abrasion, not only to remove the so-called dense smear layer to provide higher permeability, but also to remove  $Al_2O_3$  debris left on the dentin surface, since these particles may influence the resin monomer infiltration, as seen with the results of this study [27].

*In vitro* studies examining the bond strength of restorative materials are important because they can predict their clinical behavior and long-term success. Therefore this study also aimed to analyze the effect of surface roughness on the microshear bond strength of nanofilled and nanohybrid composite resin using self etchant adhesives on dentin. Group 1A (Bur + Nanohybrid) showed relatively higher mean Microshear bond strength as compared to Group 1B (Bur + Nanofilled). Group 2A (Air abrasion + Nanohybrid) also showed significantly higher mean micro tensile bond strength as compared to Group 2B (Air Abrasion + Nanofilled) at  $P=0.04$ .

The results infer that Group 1A showed significantly higher mean Microshear bond strength, followed by Group 1B, Group 2A & least with 2B. (Graph no.2). The results of the present study inferred that Nanohybrid composite yielded better bond strength than the Nanofilled composites.

Nanohybrid resin composites improve the distribution of fillers in the matrix by combining nanoparticles with submicron particles to achieve better mechanical, chemical, and optical properties. The Nanohybrid composite used here was reinforced with zirconia fillers. Nano-zirconia particles have been added to resin composites as reinforcing or toughening components due to their unparalleled mechanical strength [28]. They show effectiveness at enhancing flexural strength and wear resistance. Several studies have concluded that commercially available Nanocomposite materials do not hold any significant advantage over hybrid composites in terms of strength and hardness [29-31].

Several strategies such as micro-tensile and micro-shear bond strength tests may be used to evaluate and characterize dentin adhesion. In this study, micro-shear bond strength test was used to evaluate bond strength to dentin. Micro-shear bond test, although possessing some limitations, remains useful as screening tools for new dental materials, adhesive approaches, and investigation of different experimental variables.

Reliable and accurate measurements of the microshear bond test can be achieved if only the adhesive failures are considered for the bond strength calculation, which requires microscopic evaluation to verify the failure mode, and these requirements were fulfilled in the present study. reliability of bond strength data depends on a

number of adhesively failed specimens. In the present study, Adhesive failure at interface was more predominant as compared to cohesive failure, ranging between 60 – 80%(Graph no.3). Group 2A demonstrated relatively increased percentage of cohesive failure in resin [40%] as compared to other groups [20%]. Cohesive failure in dentine was only seen in Group 1A & Group 2B with 20% each.

Although some studies stated that air abrasion does not lower the dentin bond strength, the present study denoted that, air abrasion with  $Al_2O_3$  particles significantly lowered the micro-shear bond strength of the composite to dentin following the application of self etch adhesive. Regardless the fact that the surface roughness obtained with air abrasion did not enhance the bond strength in the present study, it is possible to infer that this characteristic is not the only factor to influence adhesion, physical parameters and the chemical composition of dentin substrate also influence adhesion.

## 5. CONCLUSION

This invitro study investigated the combined effect of  $Al_2O_3$  air abrasion and self etch adhesive on the microshear shear bond strength of nanocomposite to dentin using universal testing machine. Within the limitations of this study it could be concluded that Air abrasion decreased microshear bond strength to dentin. The self etch adhesive used could not completely dissolve the dense and amorphous smear layer produced by air abrasion, causing limited penetration of resin monomers into the dentinal tubules, that formed an ill-defined and disrupted hybrid layer which in-turn led to low microshear bond strength. Further *in-vitro* and *in-vivo* studies with larger sample sizes are required to validate the use of air abrasion on dentin and substantiate its effect on surface roughness, bond strength with various restorative materials, compatibility of air abrasion with various adhesive systems, and safety in using air abrasion on a regular basis.

## CONSENT AND ETHICAL APPROVAL

It is not applicable.

## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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