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Original Research

A Comparison of Shear Bond Strength of New Nanofilled Composite and Nano-Ionomer Restorative Materials with Traditional Adhesive Material for Orthodontic Bracket Bonding: An *In Vitro* Study

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Abstract:

Background: New materials that are introduced in operative dentistry can potentially have orthodontic applications. One of the most important advances in the dental material field is the application of nanotechnology to resin composites. Recently, new fillers with size ranging from approximately 5-100 nm have been developed. These materials could thus be considered as precursors of nanocomposites. Due to the reduced dimension of the particles and to a wide size distribution, an increased filler load can be achieved that reduces polymerization shrinkage and increases mechanical properties such as tensile and compressive strength and resistance to fracture. As new adhesives, composite resins, and bonding techniques are introduced, orthodontists adopt some of these innovations and add them to their armamentarium. The purpose of this study was to compare the shear bond strength (SBS) of the brackets bonded with the newly introduced nanocomposites with that of conventional orthodontic adhesive and to compare amount of remnant adhesive on tooth surface after debonding.

Materials and Methods: This *in vitro* study was carried out on 100 premolar teeth using conventional orthodontic bracket bonding adhesive and the newly introduced nanocomposites for bonding the 0.022 MBT Brackets (Gemini series, 3M Unitek). Teeth were randomly divided into five groups. The bonding was done according to the manufacturer's instructions followed by the curing using halogen curing unit (3M ESPE, ELIPAR, 2500). Instron Universal Testing Machine was used in this study to record the SBSs of the brackets in MPa. Following the debonding procedure, the Adhesive Remnant Index (ARI) was used to determine failure site locations.

Results: The analysis of variance test to compare SBS values of the five groups indicated statistically significant difference ($P < 0.0001$) among the groups except for Tetric N Ceram (Group III). The bond strength (MPa) is highest in Group I, followed by Group III > Group IV > Group II > Group V. Chi-square test showed no significant association between the ARI scores and the different groups. Weibull analysis showed maximum failure probability of 85% with Group V (Ketac™ N100 Group) and minimum failure probability of 25% with Group I (Transbond XT Group).

Conclusions: SBSs of restorative nanocomposites groups recorded are significantly lower when compared with conventional bonding adhesive group. Although, the bond strength values of the brackets bonded with nanocomposites is within the range of 5.9-7.8 MPa and beyond, which is clinically acceptable for effective orthodontic bonding, the probability of failure is higher for Groups II, IV, and V, which may be due to high viscosity and inability of these materials to penetrate the mesh of the bracket base. In all groups most of the material remained adhered to the tooth surface after the debonding of the brackets which is desirable. The nanocomposites and nano-ionomers can be used for bonding orthodontic brackets. However, reformulation of the composition of nanocomposites to produce better flow is desired.

Key Words: Nanofilled, nanohybrid, nano-ionomer, shear bond strength

Introduction

Since the introduction of the acid etch bonding technique by Buonocore[1] in 1955, the concept of bonding various resins to enamel is being used widely in dentistry, including the bonding of orthodontic brackets, either direct or indirect.

The adhesive systems have undergone considerable evolution after 1966, when the earliest orthodontic brackets were bonded directly onto the enamel. One of the most dramatic changes in the orthodontic specialty in the 1970s was the use of composite resin as a bonding material.[2]

The chemically cured resins were the first systems developed for bracket bonding.[3,4] Ultraviolet light-cured materials were introduced as an alternative to self-curing resins. Due to safety problems[5] and limited depth of cure,[6,7] ultraviolet light curing has been replaced by visible light curing and this has become the most popular method of polymerizing orthodontic adhesives.

When bonding an orthodontic bracket, the bond strength should be sufficient to withstand the forces of mastication and stresses exerted by arch wires. However, there are many factors that contribute to the bond strength between enamel and the orthodontic bracket, including the type of enamel conditioner, acid concentration, length of etching time, composition of the adhesive, bracket base design, the bracket material, the oral environment, as well as the skill of the clinician.

Filled dental restorative materials have been used as orthodontic adhesives.[8] These materials consist of an organic diacrylate (bisphenol A diglycidylether methacrylate: BIS-GMA), a coupler (silane), and a high percentage content of inorganic filler (quartz, silica).

It is well-known that the inorganic filler makes the material more abrasion resistant, increases the shear bond strength (SBS), and decreases the coefficient of thermal expansion to values closer to those of enamel to prevent long-term microleakage.[8,9]

One of the most important advances in the dental material field is the application of nanotechnology to resin composites. Nanotechnology can be broadly defined as, "The creation, processing, characterization, and utilization of materials, devices, and systems with dimensions on the order of 0.1-100 nm, exhibiting novel and significantly enhanced physical, chemical, and biological properties, functions, phenomena, and processes due to their nanoscale size."[10]

The recently introduced nanocomposites have been produced by nanofiller technology and formulated with nanomers and nanocluster filler particles.

Nanomers are discrete nanoagglomerated particles of 20-75 nm size, and nanoclusters are loosely bound agglomerates of nano-sized particles.

The combination of nanomer-sized particles and nanocluster formulation reduces the interstitial spacing of the filler particles and, therefore, provides increased filler loading that reduces polymerization shrinkage and increases mechanical properties such as tensile and compressive strength and resistance to fracture.[11,12]

Demineralization of the labial surfaces of teeth during orthodontic therapy is of clinical importance[13] and may present an esthetic problem, even more than 5 years after treatment.[14] One of the most effective agents in caries prevention is fluoride. It inhibits the metabolism of the bacteria that cause caries and also increases the resistance of enamel and dentine.

The importance of fluoride use during treatment with fixed orthodontic appliances to prevent development of white spot lesions is emphasized.[15] Usually, the fluoride is applied as solutions, pastes, or varnishes designed for the whole dentition.

Because of the anticariogenic and re-mineralizing effects, resin-modified glass ionomer cements (RMGIC) can be used where a locally strong initial fluoride effect is desired in addition to a long-term effect.[15]

Recently, a new RMGIC (Ketac™ N100) has been introduced for operative dentistry which is a dual curing nano-ionomer and includes fluoroaluminosilicate glass, nanofillers, and nanoclusters combined to improve mechanical properties.[16] It shows high fluoride release that is rechargeable after being exposed to a topical fluoride source.

Today, there are several nanocomposite materials available in the market. Therefore, this study was undertaken to compare the SBS of these newer materials (Ceram-X [Dentslpy], Filtek Z350 XT [3M ESPE], Tetric N Ceram [Ivoclar Vivadent], Ketac™ N100 Light Curing Nano-Ionomer [3M ESPE]) by the different manufacturers with that of Transbond XT, 3M Unitek, Monrovia, CA, USA (the Conventional orthodontic bracket bonding composite).

Materials and Methods

This *in vitro* study was conducted according to a protocol suggested by Fowler *et al.*[17] and by Fox *et al.*[18] on 100 human premolar teeth without caries or fillings that had been extracted for therapeutic purpose in patients undergoing orthodontic treatment in the Department of Orthodontics, MR Ambedkar Dental College, Bengaluru. Teeth were washed to remove any traces of blood and then stored in distilled water for up to 1-month.

One hundred stainless steel MBT pre-adjusted 0.022" slot dimension brackets (Gemini series, 3M Unitek, Monrovia, California) were used for bonding. The adhesives used were:

Polymerization source used was quartz-tungsten halogen light cure unit (Elipar 2500, 3M ESPE).

Method of collection of data

100 premolar teeth were arbitrarily divided into five groups of 20 each.

Bonding

For all groups, the buccal surfaces of premolar teeth were polished with a rubber cup and polishing paste for 30 s, rinsed thoroughly with water and dried with air stream. The area where the bracket was to be placed was etched with 37% phosphoric acid for 30 s and washed with water and dried until a frosty white appearance of the enamel was present. After acid etching, the brackets were bonded in the following manner.

Group I (Transbond XT group)

Transbond XT (3M Unitek) primer was applied to the etched surface in a thin film and then cured for 20 s. Transbond XT adhesive paste was applied to the bracket base, and the bracket was then positioned on the tooth and pressed firmly into place. The excess adhesive was removed from around the bracket with a scaler, and the adhesive was light cured from the mesial and distal sides for 20 s each (total time 40 s).

Group II (Filtek Z350 XT universal restorative group)
According to the manufacturer's instructions, the primer (Adper adhesive systems; 3M ESPE, Seefeld, Germany) was applied to the etched surface in a thin film and light cured for 10 s. Following the application of the composite, the bracket was then positioned (Filtek Z350 XT universal restorative, 3M ESPE) and cured for 40 s.

Group III (Tetric N Ceram group)
According to the manufacturer's instructions, the primer (Tetric N Bond [Ivoclar Vivadent]) was applied to the etched surface in a thin film and light cured for 10 s. The bracket was then positioned, after the application of the composite (Tetric N Ceram [Ivoclar Vivadent, Schaan, Liechtenstein]) and light cured for 40 s.

Group IV (Ceram - X group)
According to the manufacturer's instructions, the primer (Prime and Bond NT [Dentsply]) was applied to the etched surface in a thin film and light cured for 20 s. Following the application of the composite, the bracket was then positioned (Ceram-X [Dentsply, Milford, USA]) and light cured for 40 s.

Group V (Ketac™ N100 group)
After normal tooth preparation, Ketac Nano Primer (3M ESPE) was painted over the enamel surface for 15 s. The primer was thinned using a gentle stream of dry air and cured for 10 s. The desired quantity of nano-ionomer (Ketac ™ N100) was blended with a spatula for 20 s on a mixing pad. The mixed paste was then applied onto the bracket base, and bracket positioned on the tooth, and pressed firmly into place. After the removal of excess material, the adhesive was light cured from the mesial and distal for 20 s each (total time 40 s) according to the manufacturer's instructions.

Methods

An Instron Universal Testing Machine was used in this study to record the SBS. Load was progressively applied till the bracket was debonded from the tooth surface. The load at which the bracket was debonded was recorded. Following the debonding procedure the Adhesive Remnant Index (ARI) was used to check the surface of the enamel with a magnifying lens of magnification Power 5X.

Preparation of bonded tooth for SBS test

Acrylic blocks were used to mount the teeth to be subjected for SBS test. The acrylic blocks were made with self cure acrylic resin and the prepared teeth were centered in the acrylic blocks with the long axis of the teeth perpendicular to the base of the block. Later the acrylic blocks with the teeth mounted were stored in distilled water at room temperature before subjecting to SBS test.

Testing of SBS

The SBS test was conducted in the laboratory at 3M, Bengaluru. The bond strength in shear mode was recorded using an Instron Universal Testing Machine. Acrylic blocks were positioned in the Instron Machine with the long axis parallel to the direction of the load application. A stainless steel wire 0.5 mm diameter in the shape of a loop was fixed to the upper cross head at one end and was adjusted to engage the bracket at the other end. A load side density of 0-50 kg was set in the Instron Machine and the cross head speed was adjusted for 5 mm/min. An occlusal gingival load was progressively applied till the bracket was debonded from the tooth surface. The load at which the bracket debonded was calculated in Mega Pascals.

Following the debonding procedure, the surface of the enamel from which the bracket was debonded was examined using magnifying lens of magnification power 5X to assess the residual adhesive on the debonded tooth surface. The residual adhesive remaining on the teeth was assessed by using the ARI, as described by Artun and Bergland. The remaining adhesive was scored with respect to the amount of resin material that remained on the surface of the tooth:
Score 0: No adhesive remained on the tooth
Score 1: Less than 50% of the adhesive remained on the tooth
Score 2: More than 50% of the adhesive remained on the tooth
Score 3: All adhesive remained on the tooth.

The ARI scores were used to assess the sites of bond failure on the enamel-adhesive interface and the adhesive bracket interface.

Results

Following this procedure, the mean and standard deviation was calculated for each group for statistical evaluation of experimental data. Comparison of the means of SBS values was made with analysis of variance (ANOVA). Multiple comparisons were undertaken using *post-hoc* Tukey test. The results of the SBS were subjected to Weibull analysis (survival analysis), which facilitates comparison between the groups. Chi-square test was used to determine significant differences in the ARI scores between the groups.

The difference in bond strength between the groups was assessed by one-way ANOVA test (Table 1 and Graph 1).

One-way ANOVA test revealed that the different groups exhibited different bond strengths which were statistically different.

Group I showed highest bond strength compared with other groups. The bond strength (MPa) is higher in Group I (9.97) followed by Group III (8.64) > Group IV (7.94) > Group II (7.40) > Group V (6.28).

Following ANOVA, Pair wise multiple comparison between the groups was done using Tukey's *post-hoc* test by determining

Table 1: ANOVA.

Source of variation	Sum of squares (SS)	Degree of freedom (df)	Mean sum of square (MS)	Variance of ratio (*F* value)	*P* value
Between groups	152.07	4	38.02	10.09	<0.0001
Within groups	357.88	95	3.77		
Total	509.95	99			

ANOVA: Analysis of variance

honest significant difference (HSD) value, which were found to be HSD (0.05) = 1.71 and HSD (0.01) = 2.06 for the difference between the groups.

Analysis of ARI scores (Table 2 and Graph 2)

Chi-square test was used to find out if there was any significant association between the ARI scores and the different groups.

No statistically significant association was found between the ARI scores and the groups ($P > 0.05$).

Weibull analysis was done to evaluate probability of failure of premolar metal brackets (Table 3).

Weibull analysis shows maximum failure probability of 85% with Group V (Ketac™ N100 Group) and shows minimum failure probability of 25% with Group I (Transbond XT Group) (Table 3).

Failure probability shows the following trend:

Group I < Group III < Group IV < Group II < Group V

The probability of failure has been calculated at clinically acceptable SBS of 8 MPa.

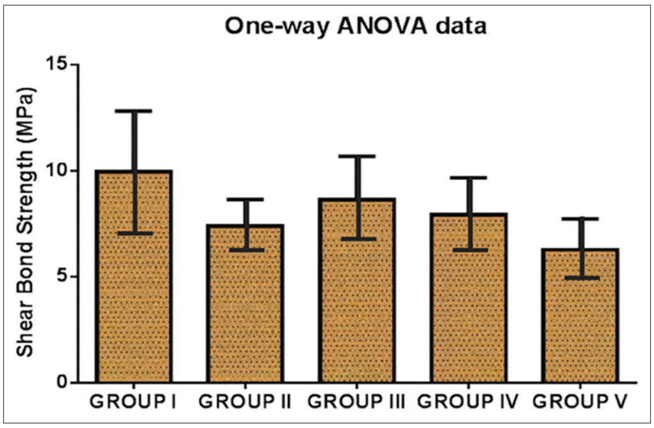
Table 2: Comparison of ARI score in samples studied.

Group	Score 0	Score 1	Score 2	Score 3	χ^2	*P* value
Group I	0	1	17	2	6.83	0.869
Group II	0	2	16	2		
Group III	1	1	18	0		
Group IV	1	1	17	1		
Group V	1	1	18	0		

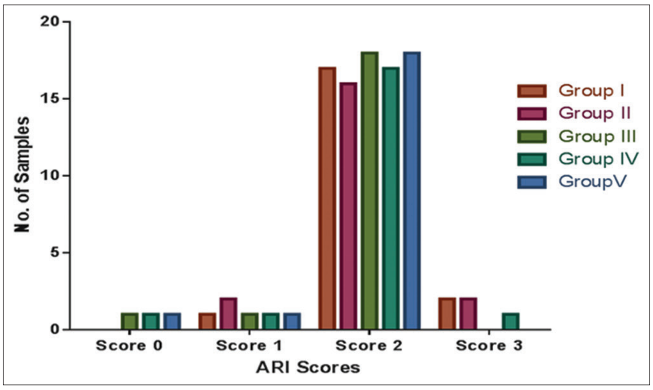
ARI: Adhesive remnant index

Table 3: Probability of failure premolar metal brackets tested at clinically accepted shear bond strength of 8 MPa.

Group	Number of teeth	Bonding material used	Probability of failure (%)
Group I	20	Transbond XT	25
Group II	20	Filtek Z350 XT	70
Group III	20	Tetric N Ceram	35
Group IV	20	Ceram-X	50
Group V	20	Ketac™ N100	85



Graph 1: Mean shear bond strength recorded in all the groups.



Graph 2: Adhesive Remnant Index scores recorded in all the groups.

Discussion

Bonding of orthodontic brackets has become a routine procedure in fixed appliance therapy as it has several advantages such as enhanced ability for plaque removal by the patient, minimization of soft-tissue irritation, elimination of the need for separation, absence of post-treatment band spaces, facilitation of application of attachments to partially erupted teeth, minimization of the danger of decalcification with loose bands, easier detection and treatment of caries, and a much more esthetic appearance for the patient.[19] A successful bracket adhesive must have adequate SBS for its continued attachment during the required clinical period.

Dental composites are composed of four chemically different materials: organic matrix or organic phase, inorganic matrix i.e., filler or dispersal phase, the initiator accelerator system, and the organosilane or coupling agent to bond the filler to the organic resin. Bis-GMA is the most commonly used monomer in contemporary composites. The dispersal phase of composite resins is made up of an inorganic filler material that, in essence, determines the physical and mechanical properties of the composite. New studies have been carried out to evaluate the influence of filler level on the bond strength of orthodontic adhesives. It has been realized that addition of the fillers to the polymeric part of the adhesives results in increased strength, stiffness, reduced dimensional changes, and improved handling characteristics.[20,21]

At present, most composites are filled with silicate particles based on oxides of barium, strontium, zinc, aluminum, or zirconium. In spite of the great variety, there is no superiority of any specific filler because every type of filler offers advantages and disadvantages. However, the best mechanical properties could be achieved by incorporating high concentrations of filler particles of various sizes into the resin.[21] Most bonding studies use commercially available adhesive systems that have different particle sizes, viscosities, and concentrations of filler particles. This makes comparisons among studies difficult because of the increased number of variables involved in the material composition. However, it has been found in various studies that there is an increase in shear and torsional bond strengths with increasing concentrations of adhesive filler.[20,22]

Resin composites have usually been classified according to filler features, such as type, distribution or average particle size. In addition to traditional microhybrid and microfilled materials, nanofilled and nanohybrid composites were more recently introduced in an endeavor to provide improved properties like increased bond strength, decreased dimensional changes and high initial polishing combined with superior polish and gloss retention.[23]

It is known that the shape, amount and size of the particles reinforcing the composite might affect their properties;[24] finer particle size results in less interparticle spacing, more protection of the softer resin matrix and less filler plucking.[25]

In a study it was concluded that the nanohybrid resins generally presented inferior properties compared with the nanofilled composite and either similar or slightly better properties compared to the microhybrid material. Under clinical conditions, nanohybrid resins may not perform similarly to nanofilled materials.[23]

It was suggested that a minimum bond strength of 5.9- 7.8 MPa is adequate for most orthodontic needs during routine clinical use.[26] Highly filled orthodontic adhesives with 80% fillers content, such as Transbond XT, provide adequate bond strength.

In a study, it was found that Bis-GMA/TEGDMA composite with filler load of 60% per by weight can be used for bonding orthodontic brackets without an intermediary low-viscosity resin while reducing the working time and the possibility of fluid contamination when compared with conventional adhesive, Transbond XT.[27]

One of the main objectives of this study was to test various newly introduced restorative materials, filled with nanoparticles that were reported to have higher physical and mechanical properties, in comparison with a conventional light-cure orthodontic bonding adhesive.

In a study, it was indicated that the nanohybrid composite system, Grandio (VOCO), achieved SBS values that were not significantly different from those obtained with Transbond XT.[28] The SBS results of this study are contrary to those findings, in that, the conventional orthodontic adhesive system showed higher values than the nano-composite and this difference was statistically significant except for the case of Tetric N Ceram (Group III).

The present *in vitro* study compared both the nanofilled as well as nanohybrid composites with the conventional orthodontic bracket bonding adhesive Transbond XT. Nanofilled composite Filtek Z 350 XT (Group II) has filler loading of 72.5% by weight.[12] Nanofilled GIC Ketac™ N100 (Group V) is claimed to have filler content of 69% by weight.[29] Nanohybrid composite Tetric N Ceram[30] (Group III) reportedly has filler content of 79.5% whereas Nanohybrid composite Ceram X (Group IV) has filler load of 76% by weight.[31] All these groups were compared to the highly filled conventional orthodontic adhesive Transbond XT (Group I), which has filler load of 80% by weight.

This study showed the SBSs of all groups which were: Group I = 9.97 ± 2.84 Mpa, Group II = 7.40 ± 1.24 MPa, Group III = 8.64 ± 2.03 MPa, Group IV = 7.94 ± 1.73 and Group V = 6.28 ± 1.45 MPa. These values were in accordance with the previous studies which demonstrated that bond strength is increased with the increase in filler load.[22,20]

Results showed higher SBS values for Group I (Transbond XT) and Group III (Tetric N Ceram) as compared to Group II (Filtek Z 350), Group IV (Ceram X) and Group V (Ketac™ N100 Nano RMGIC).

The anticariogenic and re-mineralizing effects of continuous fluoride release from conventional glass ionomer cements[32] can be predicted and there are also indications of a similar effect with RMGIC. Ketac N100 nano-ionomer was evaluated in this study, which according to the manufacturers shows high fluoride release and is rechargeable after being exposed to a topical fluoride source. The nano-ionomer did not have the disadvantage of the nano-composite wherein the consistency of the adhesive paste is thick, and the nanoionomer easily flowed into the retention pad of the bracket base. The flowability of the nano-ionomer may make it superior to composite resins for penetrating the bracket retention features and possibly coating the enamel during the bonding procedure. Such an attribute might reduce the possibility of caries forming under brackets during treatment.

In this study, ARI scores were not statistically different ($P = 0.869$) between the groups. This is in accordance with the previous study.[16] The analysis of ARI indicated that most specimens of the groups showed ARI scores of 2 followed by 3 and 1.

In all groups, most of the adhesive remained adhered to the tooth surface after the debonding of the brackets. This was independent of the adhesive used, suggesting that the bond failure was cohesive in nature (within the adhesive). This is contrary to the previous study which showed ARI score 1 for most of the specimens[16] with all the groups showing ARI score 1 indicating bond failure at adhesive and enamel interface.

Analyzing bracket debonding, it is safer and hence desirable that the failure occurs between the bracket and the adhesive or at the adhesive interface. Failure between adhesive and enamel can create enamel fractures or cause irregularities. In that way, neither the conventional composite resin (Transbond XT) nor the newer nanocomposites and nano-ionomers used in this study would cause damage to tooth surface because most failures occurred within the adhesive and few between bracket and adhesive, reducing the chances of enamel fracture. On the other hand, there was a greater difficulty in removing excessive adhesive.

The mean SBS values in all groups were all beyond the range of 5.9-7.8 MPa which is clinically acceptable for effective orthodontic bonding. The Weibull survival analysis (survival analysis) was done to predict the number of bonds likely to fail at a clinically acceptable strength of 8MPa.Weibull analysis is a survival analysis which has the ability to provide reasonably accurate failure analysis and failure forecasts with extremely small samples. Small samples also allow cost effective component testing. This analysis can be used even with inadequacies in the data.

Results showed maximum failure probability (85%) in Group V (Ketac™ N100) followed by Group II (Filtek Z 350 XT) with failure probability of 70% and Group IV (Ceram-X) with failure probability of 50% respectively. Group I (Transbond XT) showed the minimum failure probability of bonds (25%) and the values for Group III (Tetric N Ceram) were slightly higher (35%). This indicates that although the SBS values of all the materials were within the clinically accepted range, the probability of failure is higher for Groups II, IV and V which may be due to high viscosity and inability of these materials to penetrate the mesh of the bracket base.

Thus nanofilled, nanohybrid composites and nano-ionomers can be used for bonding orthodontic brackets. However, reformulation of the composition of nanocomposites to produce better flow is desired. Fluoride release from nano-ionomer might also reduce the possibility of caries formation near the interface bonding material/enamel/oral environment line. However it should be kept in mind that laboratory studies are a valuable screening tool, but clinical validation is necessary before any product or technique is universally accepted. Further studies are needed to evaluate the long term bond strength. Clinical studies are needed to validate the preliminary *in vitro* performance of the newer nanocomposite restorative materials *in vivo*.

Conclusion

This study compared the SBS of the brackets bonded with the newly introduced nanocomposites with that of conventional orthodontic adhesive. The nanocomposites used were nanofilled composite Filtek Z350 XT, nanofilled Glass ionomer cement Ketac™ N100, nanohybrid composites Tetric N Ceram and Ceram-X. It can be concluded that:

- There is a statistically significant difference in SBS values between nanocomposite groups and conventional adhesive except for Tetric N Ceram group. Nanocomposites displayed lower bond strength than the conventional adhesive. However, bond strength achieved by all the composites used was within the range previously suggested for clinical acceptability.
- There is no statistically significant difference among the nanocomposites and nano-ionomer except for Tetric N Ceram group.
- All nanocomposites except Tetric N Ceram showed high probability of failure which may be due to inability of these materials to penetrate the mesh of the bracket base. Thus the reformulation of the composition of nanocomposites to produce better flow is desired.
- The SBS is dependent on filler content i.e. it increases with the increase in filler load.
- In all groups, most of the adhesive remained on the tooth surface after the debonding of the brackets which is desirable as failure between adhesive and enamel can create enamel fractures or cause irregularities.

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