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Are nano-composites and nano-ionomers suitable for orthodontic bracket bonding?

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SUMMARY The aim of this study was to test nano-composite (Filtek Supreme Plus Universal) and a newly introduced nano-ionomer (Ketac™ N100 Light Curing Nano-Ionomer) restorative to determine their shear bond strength (SBS) and failure site locations in comparison with a conventional light-cure orthodontic bonding adhesive (Transbond XT). Sixty freshly extracted human maxillary premolar teeth were arbitrarily divided into three equal groups. The brackets were bonded to the teeth in each group with different composites, according to the manufacturers' instructions. The SBS values of the brackets were recorded in Megapascals (MPa) using a universal testing machine. Adhesive remnant index scores were determined after failure of the brackets. The data were analysed using analysis of variance, Tukey honestly significant difference, and chi-square tests.

The results demonstrated that group 1 (Transbond XT, mean: 12.60 ± 4.48 MPa) had a higher SBS than that of group 2 (nano-composite, mean: 8.33 ± 5.16 MPa; $P < 0.05$) and group 3 (nano-ionomer, mean: 6.14 ± 2.12 MPa; $P < 0.001$). No significant differences in debond locations were found among the three groups. Nano-composites and nano-ionomers may be suitable for bonding since they fulfil the previously suggested SBS ranges for clinical acceptability, but they are inferior to a conventional orthodontic composite.

Introduction

Since the introduction of the acid-etch bonding technique, the concept of bonding various resins to enamel surfaces has developed applications in all fields of dentistry, including the bonding of orthodontic brackets (Bishara *et al.*, 2007).

In routine orthodontic practice, it is essential to obtain a reliable adhesive bond between an orthodontic attachment and tooth enamel. Filled dental restorative materials have been used as orthodontic adhesives (Eversoll and Moore, 1988). 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 (Venhoven *et al.*, 1996). Ostertag *et al.* (1991), in an experimental study to evaluate the influence of adhesive filler concentration on bond strength, found an increase in shear and torsional bond strengths with increasing concentrations of adhesive filler.

One of the most important advances in the dental material field is the application of nanotechnology to resin composites. Nanotechnology is the production and manipulation of materials and structures in the range of about 0.1–100 nm by various physical or chemical methods (Kirk *et al.*, 1991). While the size of the filler particles lies between 8 and 30 μm in hybrid composites and 0.7 and 3.6 μm in microhybrid composites (Venhoven *et al.*, 1996), recently, new fillers with size ranging from approximately 5–100 nm have been developed (Moszner and Klapdohr, 2004). These materials could thus be considered as precursors of nanofilled composites. 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 (Moszner and Salz, 2001) and increases mechanical properties such as tensile and compressive strength and resistance to fracture. Geraldeli and Perdigao (2003) reported that nano-composites had a good marginal seal to enamel and dentine compared with total-etch adhesives.

Demineralization of the labial surfaces of teeth during orthodontic therapy is of clinical importance (Gorelick *et al.*, 1982) and may present an aesthetic problem, even more than 5 years after treatment (Øgaard, 1989). One of the most effective agents in caries prevention is fluoride. It will inhibit the metabolism of the bacteria that cause caries and also will increase the resistance of enamel and dentine. Forsten (1998) emphasized the importance of fluoride use during treatment with fixed orthodontic appliances to prevent development of white spot lesions. Usually, the fluoride is applied as solutions, pastes, or varnishes designed for the whole dentition (Forsten, 1998). 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 (Forsten, 1998).

Recently, a new RMGIC has been introduced for operative dentistry. Ketac™ N100 light curing nano-ionomer includes fluoroaluminosilicate glass, nanofillers, and nanofiller 'clusters' combined to improve mechanical properties. The nanofiller components also enhance some physical properties of the hardened restorative. The manufacturer calls the new restorative cement a 'nano-ionomer' because the formulation is 'based on bonded nanofiller technology'. The manufacturer has suggested that Ketac N100 shows high fluoride release that is rechargeable after being exposed to a topical fluoride source. Additionally, *in vitro* tests showed that Ketac N100 has the ability to create a caries inhibition zone after acid exposure.

No research has been published in the literature that has evaluated and compared the SBS values and adhesive remnant index (ARI) scores of orthodontic brackets bonded with a nano-composite, a nano-ionomer, and a conventional orthodontic adhesive.

The aim of this study was to test nano-filled composite and ionomer to determine their SBS values in comparison with a conventional bonding adhesive and the site of bond failure of those materials. The null hypotheses to be tested were that there is no statistically significance in (1) the bond strength and (2) the failure site location of nano-composites, nano-ionomers, and conventional orthodontic adhesives.

Materials and methods

Ethical approval for this research was obtained from the regional committee of Erciyes University.

Sixty non-carious human maxillary premolars, extracted for orthodontic reasons, were used in this study. Teeth with hypoplastic areas, cracks, or irregularities of the enamel structure were excluded. The criteria for tooth selection dictated no pre-treatment with chemical agents such as alcohol, formalin, or hydrogen peroxide. The extracted teeth were stored in distilled water until use. The water was changed weekly to avoid bacterial growth. The sample was arbitrarily divided into three equal groups. Each tooth was mounted vertically in a self-cure acrylic block so that the crown was exposed. The buccal enamel surfaces of the teeth were cleansed and polished with non-fluoridated pumice and rubber prophylactic cups, washed with water, and dried.

A 37 per cent phosphoric acid gel (3M Dental Products, St Paul, Minnesota, USA) was applied to the premolars for 15 seconds. The teeth were then rinsed with water for 30 seconds and dried with an oil-free source for 20 seconds, until a frosty white appearance of the enamel was present. Ceramic brackets (3M Unitek, Monrovia, California, USA) were bonded to the teeth, according to the manufacturer's instructions. The average surface of the ceramic bracket base used was measured and recorded as 14.54 mm^2.

After acid etching, the brackets were bonded in the following manner:

Group 1 (control group): Transbond XT (3M Unitek) primer was applied to the etched surface in a thin film and left uncured. Transbond XT adhesive paste was applied to the bracket base, and the bracket was 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 for 20 seconds each (total time 40 seconds).

Group 2 (nano-composite 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 seconds. The bracket was positioned, and the composite (Filtek Supreme plus universal restorative, 3M Espe) was light cured for 40 seconds.

Group 3 (nano-ionomer group): After normal tooth preparation, Ketac Nano Primer (3M Espe) was painted over the enamel surface for 15 seconds. The primer was thinned using a gentle stream of dry air and cured for 10 seconds. The desired number of 'clicks' of nano-ionomer (Ketac™ N100) was squeezed onto a mixing pad and blended with a spatula for 20 seconds. The mixed paste was carefully injected using an orange AccuDose syringe (Centrix, Inc., River Road Shelton, Connecticut, USA) onto the bracket base, positioned on the tooth, and pressed firmly into place. The excess material was removed from around the bracket with a scaler, and the adhesive was light cured from the mesial and distal for 15 seconds each (total time 30 seconds) according to the manufacturer's instructions.

A quartz–tungsten halogen light unit (Hilux 350, Express Dental Products, Toronto, Canada) with 10 mm diameter light tip was used for curing the specimens.

Debonding procedure

After completion of the procedures, the embedded specimens were secured in a jig attached to the base plate of a universal testing machine (Hounsfield Test Equipment Ltd., Salford, Lancashire, UK). A chisel-edge plunger was mounted in the movable crosshead of the testing machine and positioned so that the leading edge was aimed at the enamel/adhesive interface. A crosshead speed of 0.5 mm/minute was used, and the maximum load necessary to debond the bracket was recorded. The force required to remove the brackets was measured in Newtons (N), (1 MPa = 1 N/mm^2) and the SBS was then calculated by dividing the force values by the bracket base area (14.54 mm^2).

Residual adhesive

After debonding, all teeth and brackets were examined under a stereomicroscope (SZ 40, Olympus, Tokyo, Japan) at ×10 magnification. The amount of adhesive remaining on the enamel surface was coded using the criteria proposed in the ARI of Årtun and Bergland (1984).

Statistical analysis

All statistical analyses were performed using the Statistical Package for Social Sciences (SPSS for Windows 13.0, SPSS, Chicago, Illinois, USA). Descriptive statistics, including the mean, standard deviation (SD), and minimum and maximum values were calculated for the three groups of teeth tested. The Shapiro–Wilks normality test and the Levene variance homogeneity test were applied to the SBS data. The data showed normal distribution, and there was homogeneity of variances among the groups. Comparisons of the means of SBS values were made with analysis of variance (ANOVA). Multiple comparisons were undertaken using Tukey honestly significant difference tests. The chi-square test was used to determine significant differences in the ARI scores among the three groups.

Results

Descriptive statistics including the mean, SD, and minimum and maximum values for each of the three groups are presented in Table 1. ANOVA revealed statistically significant differences in bond strengths among the various groups ($P < 0.001$). Thus, the first null hypothesis of this study was rejected. The results demonstrated that group 1 (Transbond XT, mean: 12.60 ± 4.48 MPa) had higher SBS values than group 2 (nano-composite, mean: 8.33 ± 5.16 MPa; $P < 0.05$) and group 3 (nano-ionomer, mean: 6.14 ± 2.12 MPa; $P < 0.001$). No statistically significant differences were found between groups 2 and 3 ($P > 0.05$).

The ARI scores for the various groups tested are listed in Table 2. The results of the chi-square comparisons indicated that there were no statistically significant differences among three groups ($P = 0.679$). Therefore, the second null hypothesis was accepted.

Discussion

An optimal tooth restoration material should mimic the structural, mechanical, and physical characteristics of dentine and enamel (Sabbagh *et al.*, 2002). Manufacturers are continuously introducing new adhesives in operative dentistry that are more reliable, i.e. stronger, adhere better, are less prone to leakage at the margins, and/or are easier to handle (Bishara *et al.*, 2007). As new materials and techniques are introduced, orthodontists adopt some of these innovations and add them to their armamentarium (Bishara *et al.*, 2007), including the use of self-etching primers, RMGIC, chlorhexidine varnishes, and different adhesives.

Most bonding studies use commercially available adhesive systems that have different particle sizes, viscosities, and concentrations of filler particles (Ostertag *et al.*, 1991). This makes comparisons among studies difficult because of the increased number of variables involved in the material composition. Ostertag *et al.* (1991) found an increase in shear and torsional bond strengths with increasing concentrations of adhesive filler. From this starting point, the aim of the present study was to test two newly introduced restorative materials, filled with nano-particles, that were reported to have higher physical and mechanical properties, in comparison with a conventional light-cure orthodontic bonding adhesive.

Bishara *et al.* (2007) indicated that the nano-filled composite system, Grandio, achieved SBS values that were not significantly different from those obtained with Transbond XT. The SBS results of the present 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. Due to the compact consistency of the adhesive paste, the findings of Bishara *et al.* (2007) that manufacturers should consider reformulating the composition of nano-composite to produce a paste with a more flowable consistency that can readily penetrate the mesh of the bracket base for orthodontic purposes is supported.

The anticariogenic and re-mineralizing effects of continuous fluoride release from conventional glass ionomer cements 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 nano-ionomer 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

Table 1 Descriptive statistics and results of analysis of variance (ANOVA) and Tukey tests comparing the shear bond strength of three groups tested ($n = 20$).

Groups tested	Mean (MPa)	SD	Minimum	Maximum	ANOVA comparisons (significance)	Nano-composite	Nano-ionomer
Control	12.60	4.48	6.43	18.57		$P = 0.017$*	$P = 0.000$***
Nano-composite	8.33	5.16	1.71	20.71	$P = 0.000$***		$P = 0.324$, NS
Nano-ionomer	6.14	2.12	2.43	10.00			

SD, standard deviation; NS: not significant; *$P < 0.05$; ***$P < 0.001$.

Table 2 Frequency of distribution of adhesive remnant index (ARI) scores (%) in the three groups tested.

Groups tested	ARI scores*					Significance *P* value
	ARI = 0	ARI = 1	ARI = 2	ARI = 3	*n*	
Control	0 (0%)	16 (80%)	2 (10%)	2 (10%)	20	
Nano-composite	0 (0%)	15 (75%)	4 (20%)	1 (5%)	20	0.679
Nano-ionomer	1 (5%)	13 (65%)	4 (20%)	2 (10%)	20	

*ARI scores: 0 = no adhesive remains on the tooth surface; 1 = less than half the adhesive remains on the tooth surface; 2 = more than half the adhesive remains on the tooth surface; 3 = all the adhesive remains on the tooth surface.

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. Fluoride release and recharge might also reduce the possibility of caries formation near the bonding material excess interface between the bonding material/enamel/oral environment line. This linear boundary must be of high free energy based on the free energy calculation of potentially unsatisfied valence states and fluoride ions in that area should be available to form fluorapatite from present hydroxyapatite. From this perspective, Ketac N100 nano-ionomer should be considered a potentially useful adhesive for bonding orthodontic brackets. However, the SBS value of this material was also statistically lower than orthodontic composite, while no statistically significant differences were found between the nano-composite and nano-ionomer.

Reynolds (1975) suggested that a minimum bond strength of 5.9–7.8 MPa is adequate for most orthodontic needs during routine clinical use. All mean bond strength values of the materials used in this study were above this minimum requirement and within clinically acceptable ranges. However, clinical conditions may differ significantly from an *in vitro* setting. It needs to be emphasized that this was an *in vitro* study and the test conditions were not subjected to the rigours of the oral environment. Heat and humidity conditions of the oral cavity are highly variable. Because of the probable differences *in vivo* and *in vitro*, a direct comparison cannot be made with the findings of other studies.

Bishara *et al.* (2007) indicated that 15 per cent of brackets tested in their study did not register any debonding force because of the difficulty in handling the material, contrary to the findings of the current research. Adhesive remnant data were not presented in the study of Bishara *et al.* (2007) but these probably showed similar failures between orthodontic and nano-composite, considering the SBS values that they reported. The differences in the SBS values among the three different groups are reflected in the distribution of the ARI scores (Table 2). In the present study, ARI scores were not statistically different between the groups and failures in all groups were mostly located at the adhesive/enamel interface.

Conclusions

1. The nano-composite and nano-ionomer tested in this study resulted in lower bond strength values than the conventional orthodontic adhesive but demonstrated SBSs which were within the range previously suggested for clinical acceptability.
2. There were no significant differences in the mean SBS values of the tested nano-composite and nano-ionomer.
3. No significant differences in debond locations were found among the three groups investigated.

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