



## A Comparative Study of a New Microtensile Testing Device for Dental Research

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### Authors' contributions

This work was carried out in collaboration between all authors. Authors GR, JAA and LMP designed the study, wrote the protocol, and wrote the first draft of the manuscript and managed literature searches. Author TLA performed the statistical analysis. Authors FCPG, PMY, AAM, RLP and ACA managed the analyses of the study and literature searches. All authors read and approved the final manuscript.

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### ABSTRACT

**Aims:** This study aimed to compare the performance of a new microtensile bond strength ( $\mu$ TBS) testing device, called the Flextest device.

**Study Design:** This is an experimental randomised study in which composite resin/dentin stick-

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shaped specimens were submitted to a microtensile bond strength test in both Flextest device and Bencor Multi-T device.

**Place and Duration of Study:** University of Brasília (Brasília, Brazil). The duration of this study is 2 years.

**Methodology:** Fifteen human third molars were submitted to dentin surface exposure, polishing, bonding treatment, and composite blocks that were built up and sectioned to obtain composite resin/dentin stick-shaped specimens with a cross-sectional area of approximately 0.9 mm<sup>2</sup>. One hundred composite resin/dentin specimens were assigned randomly to the Bencor Multi-T device (Danville Engineering, San Ramon, CA, USA) group (n = 50) and the Flextest device group (n = 50). Tensile bond testing was performed by a universal testing machine (MTS, Eden Prairie, MN, USA) at a crosshead speed of 1.0 mm/min. The intragroup and intergroup  $\mu$ TBS comparisons, and the ratios of the types of failure in each group were analysed by factorial analysis of variance, Fisher's test, and the Chi square test at 5% significance.

**Results:** Both devices showed significant intragroup differences. However, the differences were not significant between the devices.

**Conclusion:** The new Flextest device and the Bencor Multi-t device had similar microtensile bond strength test results; however, a lower standard deviation was observed in the Flextest device group.

*Keywords: Dentin bond strength; microtensile test; microtensile test device.*

## 1. INTRODUCTION

The microtensile bond strength ( $\mu$ TBS) test was first introduced by Sano et al. [1] to evaluate the ultimate tensile strength and modulus of elasticity of mineralised and demineralised dentin. The  $\mu$ TBS test is a common approach to evaluate the bond strength of adhesive materials to teeth. In comparison to the conventional method, the  $\mu$ TBS test is able to load multiple test specimens from each tooth so that the means and variances can be calculated for a single tooth. In addition, adhesive failures are more frequent than cohesive failures during the  $\mu$ TBS test. Therefore, the measurement of higher values of interfacial bond strengths and the testing of irregular surfaces and of relatively small areas are allowed. A drawback of the  $\mu$ TBS test is that it involves more laborious techniques.

No broad agreement can be evidenced within the scientific community as to the standard protocol, the most appropriate testing device, and the application and interpretation of these methods, although papers regarding these have been published. During the test, some factors such as adhesive materials, adhesive application methods, the geometry and dimensions of the specimens, load application, and factors inherent to the testing device may influence the microtensile bond strength test results [2-4]. The testing device performances may interfere with the cohesive microtensile values and with the tensile force distribution pattern through a specimen [5].

An ideal microtensile testing device should provide moment-free axial force application and should render accurate specimen fixation. In targeting these goals, some testing devices have been developed such as the Geraldeli device, the Andreatta Filho device, the Borges, the MT-jig, and the Bencor Multi-T device (Danville Engineering Co., San Ramon, CA, USA) [5,6]. Botta et al. [5] compared the performance of three testing devices with a customised gauge for the microtensile test, and they found that the devices differed significantly in performance and consequently resulted in discrepant microtensile bond strength values. The Bencor Multi-T device has been used broadly in several studies; however, no information is available with regard to its accuracy in comparison to other devices. Furthermore, new testing devices can be evolved with improvements in structural design, in mechanical features, and in functionality.

With this concern, a project for a new microtensile/compressive device, called the Flextest device, has been designed by the mechanical engineers of the University of Brasília (Brasília, DF, Brazil) and Mitay Industrial Precision Mechanics Company (Cotia, SP, Brazil). The Flextest device has been developed to ensure the correct positioning and fixation of the specimen and a better distribution of tensile forces to all surfaces of the specimen. This feature provides greater reliability when the Flextest device is compared to other existing devices. Moreover, the device was designed to

perform compression tests. The aims of the present study are two-fold: (1) to evaluate the viability of the Flextest device for microtensile tests and (2) to compare quantitatively and qualitatively its performance with the Bencor Multi-T device.

## 2. MATERIALS AND METHODS

The Ethics Committee of the School of Health Sciences at the University of Brasília (Brasília, Brazil) approved the present study (protocol number 078/09). All patients that agreed to participate in this study signed an informed consent statement.

For sample selection, fifteen nonfunctional partially or totally erupted human third molars were extracted and collected. The exclusion criteria were teeth with cavities, teeth with wear, or teeth with restorative materials. After extraction, the teeth were cleaned, and visually inspected under artificial light and magnification. After confirming the integrity of the dental crown and root, the teeth were immersed in 0.1% thymol solution at 4°C before submitting them to specimen preparation.

### 2.1 Specimen Preparation

The same operator prepared all specimens. A diamond saw (KG Sorensen, Cotia, SP, Brazil) mounted on a cutting device (Secotom; Struers, Ballerup, Denmark) was used to cut the teeth under running water at the midcoronal level so that flat dentin surfaces of all teeth were exposed. After the cutting procedure, the dentin surfaces were polished with wet 600-grit silicon carbide paper (Acqua Flex; Norton, Guarulhos,

SP, Brazil) for 30 seconds to generate a standardised smear layer [7]. Each tooth was thereafter submitted to the bonding treatment in accordance with the manufacturer's instructions (Table 1).

After the bonding treatment, resin composite (Z250, A2 shade; 3M ESPE, St. Paul, MN, USA) was incrementally built up in layers to produce 6-mm high blocks on the dentin surfaces. Each 2-mm incremental layer was cured under a light power density of 600 mW/cm<sup>2</sup> for 20 seconds (Curing Light XL 1500; 3M ESPE) in accordance with the manufacturer's instructions. The composite resin/tooth blocks were eventually immersed in distilled water at 37°C for 24 hours.

The teeth were individually fixed to a sectioning block by using acrylic resin. Each tooth was mounted on a hard-tissue microtome and serially sectioned in the occlusogingival direction. This action produced 0.9-mm thick slabs. Each tooth was then rotated 90°, and the serial sectioning was repeated. The resulting stick-shaped specimens were sectioned free from the root. These specimens consisted of the resin composite in the upper half and dentin in the lower half (Fig. 1).

Through these procedures, composite/dentin stick-shaped specimens with cross-sectional area of approximately 0.9 mm<sup>2</sup> were prepared. One hundred composite/dentin stick-shaped specimens were consequently obtained, and randomly assigned to two groups: the Benor Multi-T group (n = 50) and the Flextest group (n = 50). The Benor Multi-T group and the Flextest group comprised 8 teeth and 7 teeth, respectively.

**Table 1. Adhesive used in this study with the composition provided by the respective manufacturer: Adper single bond 2 is the material's trade name in South America. the same material is called adper single bond plus in the United States of America and adper scotchbond 1XT in Europe**

<b>Name (Manufacturer)</b>	<b>Composition</b>
Adper™ SingleBond 2/Plus (3M™ ESPE™, St. Paul, MN, USA)	Etching: 37% phosphoric acid gel (3M™ ESPE™, St. Paul, MN, USA) [4BT], primer and adhesive: dimetacrylates, HEMA, polyalkenoic acid copolymer, 5 nanometer silane treated colloidal silica, ethanol, water, photo initiator Etch for 15s; rinse for 10s; blot excess water; apply 2-3 consecutive coats of adhesive for 15 sec with gentle agitation; gently air thin for 5 sec and light cure for 10 sec.

*HEMA = hydroxyethylmethacrylate*

## 2.2 Microtensile Bond Strength Test

The Bencor Multi-T device (Danville Engineering Co.) has been described in other studies [8-11]. The protocols for its manoeuvre have also been standardised.

The Flextest device (University of Brasília and Mitay Industrial Precision Mechanics Company) is composed of a fixed lower platform with two parallel guiding bars to which a sliding upper platform is attached. The upper sliding platform has roller bearings that allow it to slide up and down during microtensile test without any risk of locking the platform. This characteristic differentiates this device from existing devices. Each platform has a sample-holding base, which has a 1.0-mm wide × 0.5-mm deep notch in the middle. On both sides of the notch are four screw holes. A metallic plate covers the surface of the holding base with the notch, which is secured by the four screws on both sides of the notch (Fig. 2).

The specimens of both experimental groups were gripped onto the microtensile devices with cyanoacrylate adhesive (Super Bonder Gel, Henkel Ltda., São Paulo, SP, Brazil) (Fig. 3).

In the Bencor Multi-T group, the specimens were visually aligned as parallel as possible to the long axis of the device. In the Flextest group, the specimens were placed in the device notch and, after curing the adhesive, the cover plate was positioned and screwed. The testing devices were connected to a universal testing machine (MTS; MTS Systems Corporation, Eden Prairie, MN, USA), which yielded a failure tension at a crosshead speed of 1.0 mm/min (Fig. 4). The microtensile bond strength was recorded by

MTS-Test Star II software (MTS Systems Corporation). The specimens that fractured during preparation were not included in the samples for evaluation.

After the microtensile test, each part of the specimen was carefully removed from the devices and stored in distilled water. The cross-sectional area at the failure site was evaluated under 40× magnification with a stereoscopic microscope (Metrimex in cooperation with Pzo-Labimex, Budapest-Hungary). The types of failure were classified as: adhesive (A; failure between the resin composite and the dentin), cohesive in dentin (CD), cohesive in composite (CR), or mixed type when at least two different types of failures were observed in the same specimen. After the evaluation, the frequency of each type of failure was estimated and compared.

## 2.3 Statistical Analysis

The obtained data were evaluated by standard data distribution through the Lilliefors test. The homogeneity of variance between the two devices was evaluated by using Levene's test. Once the data were in normality and in homogeneity between the devices, it was analysed by factorial analysis of variance, followed by Fisher's test (i.e., least square difference) to investigate the intragroup averages and intergroup differences.

Furthermore, the absolute number of specimens was evaluated for the fracture type in each of the devices, and were verified for the homogeneity of frequencies by using the Chi square test. All tests used in this study established a significance level of 0.05.

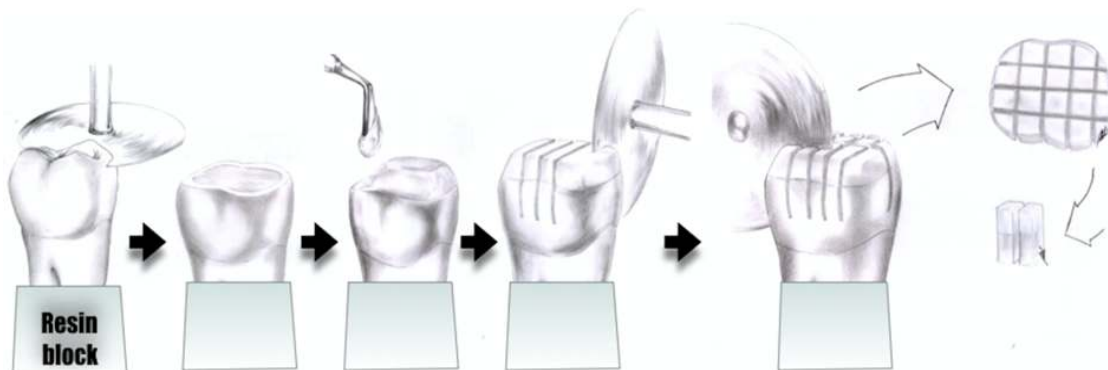
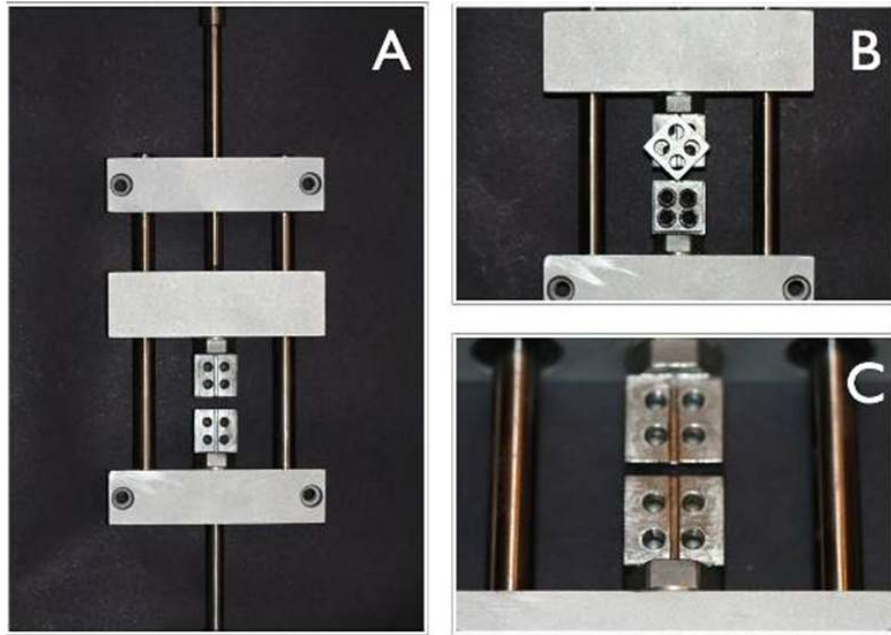
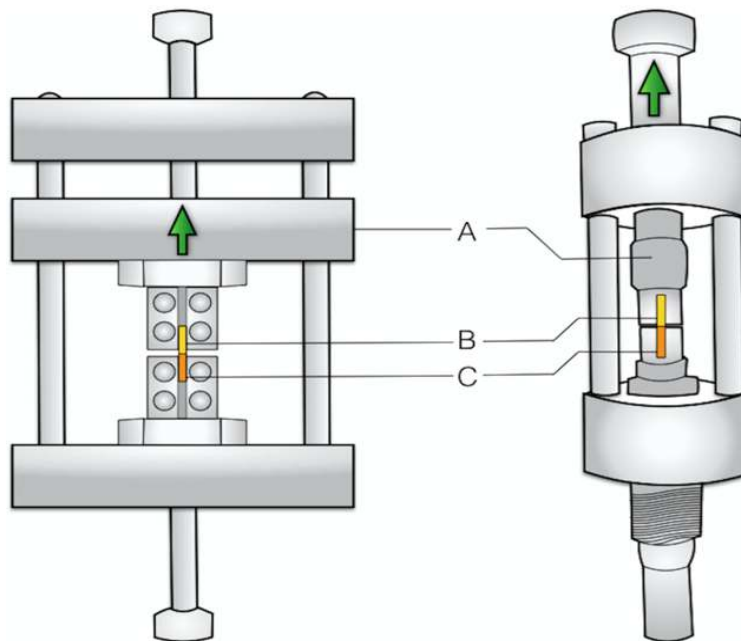


Fig. 1. Schematic of tooth sectioning and sample preparation



**Fig. 2. (A) The Flextest device. the upper platform is mobile and the lower platform is fixed. Both platforms are precisely guided by parallel bar trails. (B) the metal plate and attached screws provide an optimised tensile distribution to all surfaces of the specimen. (C) the notch is 1 mm wide and 0.5 mm deep to accommodate the specimens. the flat surface makes it possible for the mobile and fixed parts to stay in touch, which is useful for performing the compression tests**



**Fig. 3. Samples mounted in both devices. the Flextest device is on the left and the Bencor Multi-T device is on the right. (A) the sliding platforms. (B) the resin composite part of the resin/dentin stick-shaped sample. (C) the dentin part of the resin/dentin stick-shaped sample**



**Fig. 4. (A) The Flextest device mounted on the MTS universal testing machine with a load cell with a nominal capacity of 100 kg. (B) the Bencor Multi-T device mounted on the MTS universal testing machine with the same load cell**

### 3. RESULTS

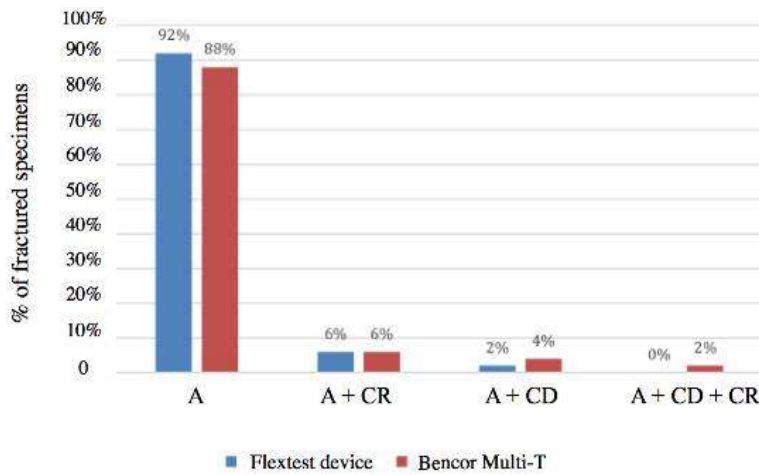
Intragroup differences were statistically significant in the Bencor Multi-T device group ( $P < 0.001$ ) and in the Flextest device group ( $P = 0.005$ ). Among the teeth submitted to the Bencor Multi-T device, it was possible to verify that tooth 4 had a significantly higher mean resistance value ( $58.31 \pm 14.11$  MPa) while tooth 8 had a lower resistance value ( $20.17 \pm 6.01$  MPa). The remaining teeth had intermediate values (Table 2). Among the teeth submitted to the Flextest device, teeth 2, 3, and 4 had significantly higher  $\mu$ TBS values ( $43.47 \pm 8.36$  MPa,  $42.13 \pm 7.76$  MPa, and  $42.13 \pm 9.62$  MPa, respectively), followed by (in decreasing value) teeth 5 ( $47.18 \pm 17.88$  MPa), 6 ( $34.69 \pm 8.17$  MPa), 1 ( $26.58 \pm 12.51$  MPa) and 7 ( $22.78 \pm 6.01$  MPa) (Table 2). In summary, the teeth subjected to both devices performed differently.

Intergroup differences were not observed ( $P > 0.05$ ). However, intragroup differences existed. The average bond strength values obtained from the Bencor-Multi-T and Flextest groups were statistically equivalent (Table 3).

**Table 2. Intragroup comparisons: The means and standard deviations of the bond strength (MPa) between teeth subjected to the Bencor Multi-T and Flextest microtensile testing devices**

Devices	Tooth	n	Mean	SD
Bencor Multi-T	1	6	26.58cd	12.51
	2	5	30.16cd	15.92
	3	7	48.38ab	11.59
	4	7	58.31a	14.11
	5	6	47.18ab	17.88
	6	6	34.69bc	8.17
	7	6	22.78cd	9.12
	8	7	20.17d	6.01
Flextest device	9	9	37.93ab	9.63
	10	8	43.47a	8.36
	11	6	42.13a	7.76
	12	6	42.13a	9.62
	13	8	29.01c	6.73
	14	7	38.61ab	6.67
	15	6	30.66bc	5.92

*Different letters indicate statistically significant differences between the means of the teeth of each device, SD = standard deviation*



**Fig. 5. The proportion of fractured specimens between the Flextest device and the Bencor Multi-T device based on the various types of fracture**

A = adhesive type of failure; CD = cohesive in dentin type of failure; CR = cohesive in composite type of failure

The two devices provided the same frequencies and homogeneity of the types of fractures ( $\chi^2 = 1.378$ ,  $P = 0.711$ ). The graphical representation is expressed in percentage (Fig. 5).

**Table 3. Comparisons between the groups: the means and standard deviations of the bond strength (MPa) of the teeth subjected to the Bencor Flextest and Multi-T microtensile testing devices**

Devices	n	Mean	SD
Bencor Multi-T	50	36.52	17.59
Flextest device	50	37.62	9.22

SD = standard deviation

#### 4. DISCUSSION

Since the introduction of the  $\mu$ TBS test by Sano et al. [1], the use of the hourglass shaped and stick-shaped specimens for the test has been broadly accepted [12-15]; however there is no consensus or protocol concerning the testing device that could be considered the gold standard. In this context, the Flextest device was designed with the main purpose of yielding more uniform tensile force distribution and serving as a multitask device. In the present study, the Bencor Multi-T device was chosen as the control device because of its broad acceptance in the literature.

In the intragroup comparisons, based on the obtained statistical data, the pattern of performance was more irregular for the Bencor Multi-T device than for the Flextest device. The frictional force of the sliding platform of the

Flextest device was minimal because of the microsphere technology of the device. This may have been a relevant aspect for the smaller standard deviation in the Flextest group than in the Bencor Multi-T group. However, it should be emphasized that the intragroup variation in the Bencor Multi-T group did not invalidate the confidence in the performance of this device. With this outcome, the intragroup variation among the mean  $\mu$ TBS values of each tooth in the Flextest group also could not invalidate this device.

The lower standard deviation and coefficient of variation of the Flextest group, compared to the Bencor Multi-T group, is likely attributable to the structural improvement added in the device. With regard to the physical structure of the Flextest device, the two guiding bars ensure the alignment and precise vertical upward and downward sliding of the upper platform. Based on this principle, a moment-free bending would be expected, and pure vertical load would be provided. With regard to the holding base, the specimen could be accurately attached in a notch with adhesive and covered by a metallic plate to distribute the tensile force uniformly in all directions. An increase of the amount of attached surfaces of the specimen to the device improves the distribution of tensile forces [4]. Further finite element method studies could be performed to check these hypotheses.

Intergroup comparison showed no significant difference between the Flextest group and the Bencor Multi-T group. This finding showed that

the Flextest device had good performance. In the present study, a lower standard deviation was observed in the Flextest group in comparison to the Bencor Multi-T group. The advantageous aspects of the Flextest device may be responsible for this result in the present study. However, this finding is not unanimous in the literature because other studies using the Bencor Multi-T device showed similar or even lower standard deviation values in comparison to the data obtained in the Flextest group [16-19].

Several studies have reported mean  $\mu$ TBS values similar to those obtained in the present study [2,16,20-22]. It should be emphasised that not only gripping devices can influence the  $\mu$ TBS test results. For instance, the cross-sectional areas of the specimens used in this study were small—approximately 0.9 mm<sup>2</sup>—and the tensile bond strength was inversely related to the bonded surface area [23]. Thus, it is not the scope of this study to discuss the merit of the different values reported in the literature in comparison to the values in the present study, but to show the similarity of the results provided by the two devices when tests were performed under similar conditions.

Concerning the type of failure, Raposo et al. [24] showed a significant association between the type of failure and gripping devices ( $P < 0.0001$ ). In the present study, this was not observed. Both devices predominantly presented a similar type of failure, which was the adhesive type, of 88% and 92% for the Bencor Multi-T group and Flextest group, respectively. Cohesive failure alone was not observed, which corroborated the statement that the microtensile test provokes fewer cohesive failures. This fact implies that the cohesive strength of the dentin tissue and the composite resin are stronger than the adhesive strength between them.

The results of the Flextest device showed that this device could be used for  $\mu$ TBS testing with high reproducibility and similarity to the Bencor Multi-T device. New features were added to correct the distribution of the tensile forces and consequently to avoid undesirable mechanical loads. The cost of production of this device is competitive. In addition, the flat surfaces of the holding bases facilitate their being in contact for the compression tests.

## 5. CONCLUSION

The Flextest device provided highly reliable and reproducible microtensile bond strength testing

performance, which showed similar mean  $\mu$ TBS values in comparison to the Bencor Multi-T group; however, a lower standard deviation was observed in the Flextest device group. The type of failure in both groups was the adhesive type.

## PATIENT CONSENT

All authors declare that written informed consent was obtained from the patient.

## ETHICAL APPROVAL

All authors hereby declare that all experiments have been examined and approved by the appropriate ethics committee and have therefore been performed in accordance with the ethical standards laid down in the 1964 Declaration of Helsinki.

## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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