Effect of Titanium Tetrafluoride on Degree of Conversion and Bond Strength of an Orthodontic Primer

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Abstract:

Objective: To evaluate the degree of conversion (DC), and orthodontic bracket shear bond strength (SBS) after the incorporation of 3 wt.% TiF4 into an orthodontic primer. **Materials and Methods:** According to the concentration of TiF4 within the orthodontic primer, there were two groups: the control (TF0C) and experimental groups (TF3C) (0&3 wt.% TiF4 respectively). The DC was evaluated for each group using FTIR. Ten orthodontic brackets were bonded using each concentration to the clean and etched enamel buccal surface. The SBS test was performed in an Instron universal testing machine, using a chisel positioned at the junction interface with a speed of 1.0 mm/min. **Results:** The DC was not significantly affected. TF3C (5.13±0.55) had the lowest SBS values in comparison to the control group (8.03±1.56). **Conclusions:** Incorporation of 3 wt.% of TiF4 within the primer of 3-step etch and rinse orthodontic adhesive was not recommended. Although it did not affect the degree of conversion, the bonding potency was greatly compromised.

Introduction:

White spot lesions (WSLs) are a significant problem in orthodontics. These are unaesthetic and unavoidable common adverse effects of orthodontic therapy with fixed appliances.[1] These areas of decalcified enamel clinically manifested as an opaque, white color lesion representing the first stage of caries formation.[2]

In addition to impairing aesthetics, the existence of untreated WSLs may cause cavity formation, necessitating additional restorative operations. Therefore, orthodontists and patients need to be aware of how to stop WSL development. The major strategies include different methods for improving enamel resistance and mechanical plaque management.

The first step in preventing demineralization is patient education and the use of fluoride in the form of paste, varnish, gel, or solution. At high concentrations, fluoride functions as a bactericidal agent, but its primary effect is to change the equilibrium of the solution to promote the development of fluorohydroxyapatite crystals.[3-5]

Fluoride treatments have been shown in numerous trials to be effective in lowering caries when used in conjunction with fixed appliances for orthodontic treatment.[6] However, the efficacy of the administration of fluoride as a preventive measure by topical applications or home rinse programs is limited due to unpredictable compliance of the patient and the fact that it is not able to produce the desired effect in the localized area adjacent to brackets.

Different orthodontic bonding agents with the capacity to release ions such as fluoride, calcium, and phosphate have been created to reduce the requirement for patient compliance, which is unpredictable and unreliable. A revolution has taken place in the area of WSLs in the recent era of orthodontic research with the evolution of novel biomimetic orthodontic adhesives prepared with the use of Titanium Tetrafluoride.

Titanium tetrafluoride (TiF4) is a fluoridated compound with low acid solubility. Due to the deposition of CaF2 and the creation of an acid-resistant titanium dioxide (TiO2) layer on the enamel, it has a substantial ability to protect against enamel demineralization.[7,8] TiF4 is a promising anticaries compound, but solutions of TiF4 alone are not stable enough to be employed in therapies, since TiF4 interacts strongly with all solutions, and has an unstable ph. Therefore, adding TiF4 to the primer or bond would make it easier to be used technically.[9]

In this study, we have known the effect of the incorporation of 3 wt.% of titanium tetrafluoride within the primer of an orthodontic adhesive on the degree of conversion and orthodontic bracket shear bond strength.

The null hypothesis was that there was no difference between the experimental orthodontic primer containing 3 wt.% of TiF4 and the commercial orthodontic primer with no TiF4 related to the degree of conversion and orthodontic bracket shear bond strength.

Materials and methods:

Ethics approval and consent to participate: The study protocol was approved by the Faculty of Dentistry, Mansoura University Ethical Committee Board of Clinical Trials and Non-interventional Research (A11040521). The Ethics Committee approved the

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informed consent dismissal. All procedures performed in studies involving human participants were following the ethical standards of the institutional and/or national research committee and with the 1964 Helsinki declaration and its later amendments or comparable ethical standards.

Materials:

1. Titanium Tetrafluoride (Aldrich Chemical Company, Milwaukee, WI, USA)
2. U-Bond orthodontic adhesive (VERICOM)
3. 3M^{TM} Scotchbond^{TM} Universal Etchant Gel
4. Orthodontic brackets (Geousis Mini Roth, ORMCO, CA, USA. Lot no: 106 120114)
5. Polishing paste (Quartz), rubber prophylaxis cups (Kerr), prophy bristle brushes (Kerr)
6. Low-speed micromotor (STRONG 210), low-speed handpiece STRONG 108E)
7. Dental prope (Dentsply), microbrushes (ELEMENT)
8. Fourier Transform Infrared Spectrometer (Thermo Nicolet iS10)
9. Universal Testing Machine (Instron, model 3345, England)

Methods:

1. **Preparation of the experimental orthodontic primer :** The commercially available TiF4 crystals were ground into powder using a mortar and pestle, weighted, and added to the etch and rinse U-Bond orthodontic primer (VERICOM) under constant agitation using a sonicator bath to achieve a 3 wt.% concentration of TiF4 within the orthodontic primer by mixing 30 mg of TiF4 with 1 mL of the primer.

2.Characterization of the prepared Tif4 primers :

Degree of conversion:

Using Fourier Transform Infrared Spectrometer (FTIR) (Thermo Nicolet iS10) (Faculty of Pharmacy, Mansoura University) with an attenuated total reflectance crystal (ATR), the degree of conversion of the dental primer containing TiF4 was assessed, Figure 1.Droplets of un-polymerized primer left unincorporated with TiF4 were used to obtain reference values corresponding to rings of the aromatic and aliphatic bands. After obtaining the reference values, the experimental primer was freshly manipulated. It was applied on a glass slide and then lightly cured for 10s taking into consideration a standard distance of 1 mm between the tip of the light unit and the sample. After that, it was covered by stretch paper and re-cured again to make sure that the maximum degree of polymerization was achieved by preventing the formation of the O2-inhibiting layer. The stretch paper was then removed, and the cured droplets were placed carefully on the crystal of the testing equipment. The degree of conversion was quantified as a summation of five repetitions for each group. Using the polymerized and un-polymerized specimens as the internal standard, the degree of conversion was estimated based on the ratio between the carbon aliphatic and aromatic double bonds. The degree of conversion was calculated by the following equation:

$$DC(\%) = \left(100 - \left(\frac{\frac{1638\,cm^{-1}}{1608\,cm^{-1}} polymerized}{\frac{1638\,cm^{-1}}{1608\,cm^{-1}} unpolymerized}\right)\right) \times 100$$

3.Selection of specimens : A total number of 20 human upper first premolars were collected from the surgery clinic (Faculty of Dentistry, Mansoura University). The teeth used for this study were collected according to the regulation of the Ethical Committee of the Faculty of Dentistry-Mansoura University. These teeth were collected from orthodontic patients whose treatment plan required extractions The teeth were inspected under normal light conditions to determine whether the teeth were appropriate for inclusion or not.

4.Enamel surface preparation:

1. A rubber prophylaxis (Kerr) connected to a slow-speed handpiece (STRONG 108E) was used to polish the labial enamel surfaces for 10 seconds with a polishing paste (Quartz, medium grit)
2. A 15-second air/water rinse followed by a 10-second oil-free compressed air stream was used to clean and dry the polished surfaces.
3. A 37% phosphoric acid solution (3M^{TM} Scotchbond^{TM} Universal Etchant Gel) was used to etch the enamel surface for 30 seconds. Following a 15-second rinse with water, the etched surfaces were dried with oil-free compressed air until the etched enamel's surface had a frosted appearance.
4. The etched premolars were divided into two groups: the control group (TF0C) with no TiF4 and the experimental group (TF3C) containing 3 wt.% of TiF4 within the orthodontic primer. Each group contained ten specimens.
5. For each specimen, to make sure that only a thin layer of primer remained, the associated orthodontic adhesive was applied with a microbrush (ELEMENT) to the etched tooth surfaces. Then, the tooth surface was softly blown with a stream of oil-free compressed air.
6. The u-Bond adhesive (VERICOM) was placed directly on the bracket base of an upper premolar series bracket with a bracket base area of 10.9 mm^2 (Geousis Mini Roth, ORMCO, CA, USA. Lot no: 106 120114). The already etched and prepared tooth surfaces were then covered with brackets.
7. Brackets were bonded to the labial surface at the intersection of the long axis of the clinical crown (LACC) and the clinical crown long axis midpoint (LA). The appropriate force was used to seat each bracket into its proper position. A pointed probe (Dentsply) was used to remove the extra adhesive, and pressure was then applied once again for five seconds.
8. Halogen light, with a wavelength of 400–500 nm and an intensity of 800 mw/cm^2 (Dentsply Spectrum 800), was used to polymerize the bonding material for 20 seconds.

5. **Bracket shear bond strength:** A universal Testing Machine (UTM) (Instron, model 3345,

Figure 1: Fourier Transform Infrared Spectrometer.

England) (Faculty of Dentistry, Mansoura University) was used to conduct the SBS test. To attach the specimens in the lower jaw of the machine so that the bonded bracket base was parallel to the direction of the shear force, they were first fixed in a plastic tube of 1-inch diameter with acrylic resin ,Figures 2&3. At a crosshead speed of 0.5 mm/min, specimens were subjected to compressive loading. A stainless-steel rod with a mono-beveled chisel configuration that was attached to the testing machine's upper movable compartment was placed precisely on the bracket base. The fracture load (F) in Newton was divided by the surface area (A) in mm^2 to determine the SBS in megapascals (MPa). By utilizing a digital caliper to measure the length and width, the bracket bonded area was estimated (Mitutoyo Corporation).[10,11]

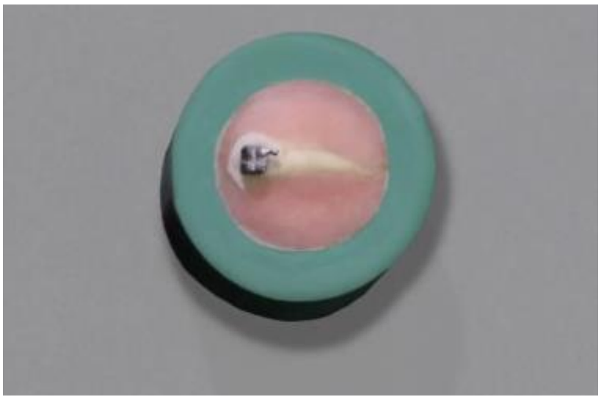

Figure 2: Preparation of Teeth for shear bond strength test

6. Statistical analysis: The normality and equal variance assumptions were fulfilled according to the Shapiro–Wilk test ($p > .05$) and Levene's test. A one-way ANOVA was conducted to analyze the DC and SBS. Tukey's significant difference tests were used for post-hoc comparisons.

Results:

1. Degree of Conversion: A comparison of the degree of conversion (DC) (%) between the control and experimental groups is shown in table 1. The results showed that there was no significant difference between the groups where ($p > 0.05$).

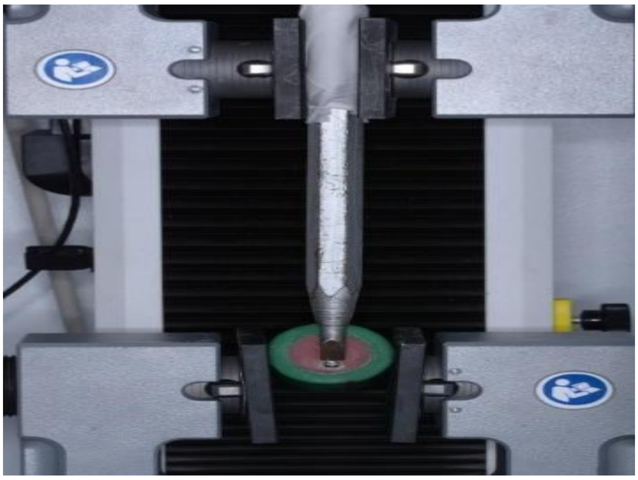

Figure 3: Universal Testing Machine (UTM).

Table 1: Comparison of the degree of conversion between different concentrations

	0%	**3%**
Degree of conversion	99.1%	99.14%
Test of significance		P0=1.0

P0: difference between the finding and 0% concentration

A graphical presentation of the absorbance peaks for the un-cured specimen of orthodontic primer without TiF_4 was shown in Figure 4 while graphical presentations of the absorbance peaks for cured specimens of dental primer without and with TiF_4 (3 wt.%) were shown in Figures 5&6. The absorbance peaks of infrared rays for the aliphatic (C = C) bond were 1638 cm^{-1} and 1637 cm^{-1} for the uncured and cured specimens. Whereas the absorbance peaks for the internal reference band aromatic C…C was 1610^{-1} and1609^{-1} for the cured and uncured specimens. **2.Bracket Shear Bond Strength (SBS) testing results:** The means ± SD of SBS values (MPa) for the two groups were represented in table 2. One-way ANOVA test showed significant differences between the two groups ($p <.001$). Tukey's significant difference test showed that the lowest SBS value was recorded for TF3C (5.13±0.55) ,Figure 7.

Discussion:

Due to the ongoing debate over TiF_4 as an enamel pretreatment and the lack of studies using conventional bonding systems, the incorporation of TiF_4 into the three-step etch and rinse primer may be crucial for the longevity of the hybrid layer and the possible inhibition of secondary carious lesions. Furthermore, the majority of investigations have reported using titanium tetrafluoride (TiF_4) in concentrations between 0.1 and 4%.[12,13] This raises the issue of what TiF_4 concentration is appropriate for clinical use to reduce enamel demineralization under orthodontic brackets. Therefore, the present study aimed to evaluate

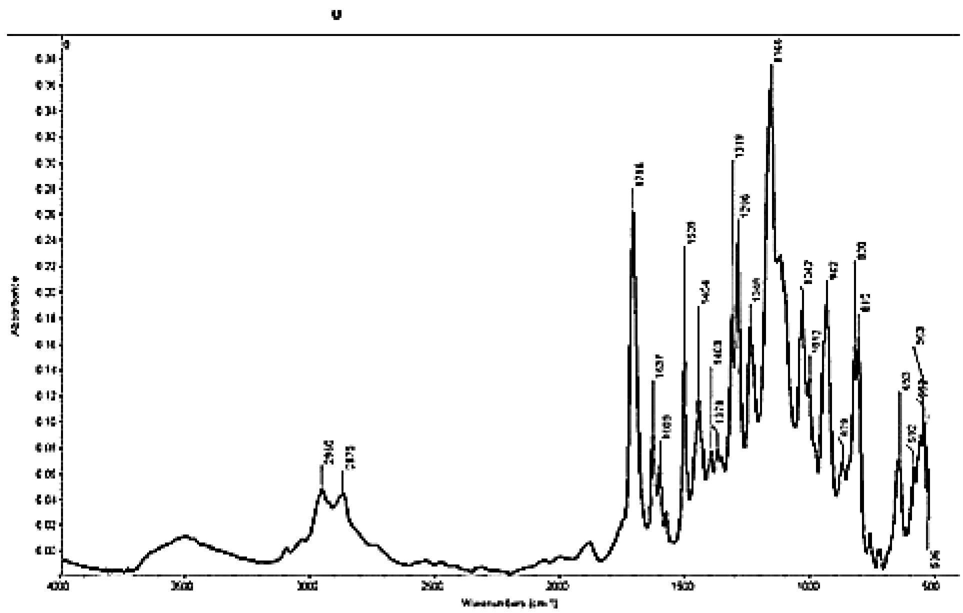

Figure 4: FTIR analysis for un-cured specimen of dental primer without TiF4. (Absorbance peak of aliphatic C = C double bond at wavelength 1637 cm^{-1} is 0.0875 while the internal reference C…C at 1609 cm^{-1} is 0.0656).

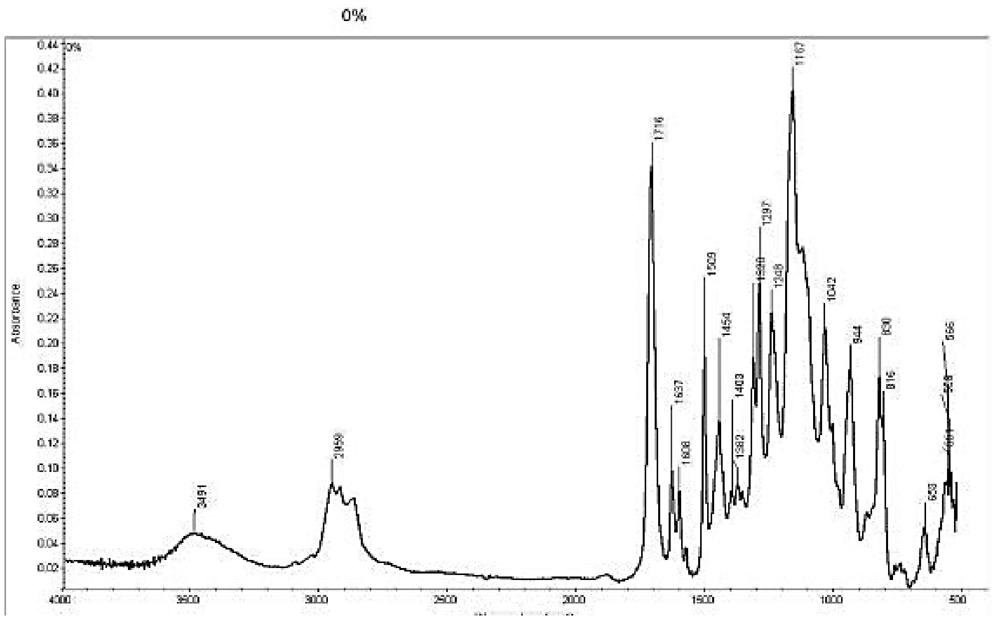

Figure 5: FTIR analysis for cured specimen of dental primer without TiF4. Absorbance peak of aliphatic C = C double bond at wavelength 1637 cm^{1} is 0.0966 while the internal reference C…C at 1609 cm^{-1} is 0.0801).

the influence of the incorporation of 3 Wt.% of TiF4 into the three-step etch and rinse primer on the degree of conversion and orthodontic bracket shear bond strength.

The most common laboratory technique for assessing the shear bond strength of brackets is shear testing.[14,15] The shearing wedge blade utilized in this investigation was a stainless-steel rod with a mono-beveled chisel configuration. This method is preferred at cross-head speeds between 0.5 and 1.00 mm/min because it permits more even stress distribution at the bonded interface and is useful, quick, and less sensitive to handling during the setting.[16] The shearing wedge was precisely positioned onto the bracket base to prevent further rotational stresses, even though the debonding forces applied in vivo are more likely to be applied to the bracket wings.[14]Metal brackets should ideally have a shear bond strength of 7-9 MPa to enamel to give adequate adhesion and make bracket removal easier once orthodontic treatment is complete.[17] The reduction in SBS for the 3% TiF4 group could be a difficult situation to explain but it might be explained

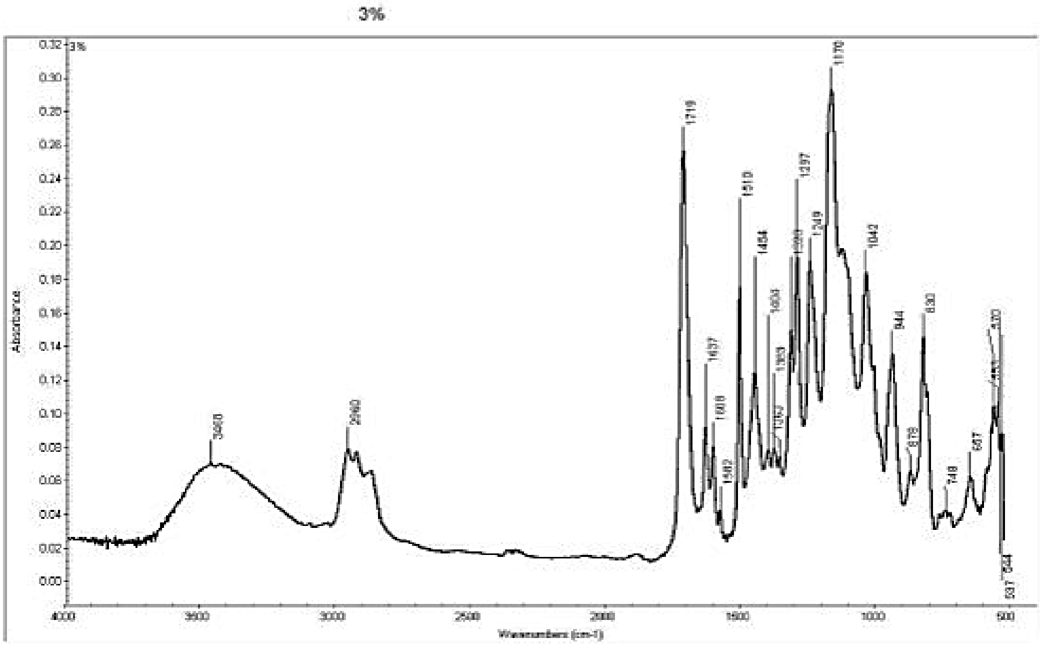

Figure 6: FTIR analysis for cured specimen of dental primer containing 3 wt.% of TiF4. (Absorbance peak of aliphatic C = C double bond at wavelength 1637 cm[1] is 0.0911 while the internal reference C…C at 1609 cm^{-1} is 0.0798).

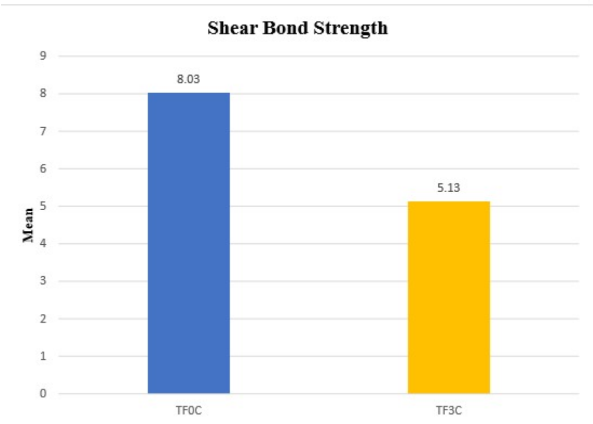

Figure 7: Graphical presentation of shear bond strength (SBS) results of the experimental groups

by two reasons; firstly, the formation of a thick CaF2 precipitated layer, which could affect bond integrity and facilitate the adhesive mode of failure[18], secondly;

the higher degree of primer's viscosity after addition of TiF4 is due to the smaller particle size of TiF4 molecule (0.55 nm) which may impede their penetration into the hybrid zone, and hence affect bonding effectiveness.[19,20] It is worth mentioning that attention must be paid to the pH of the experimental primers. The percentage size and shape of particles might also have a significant influence on the rheological properties of the primer, and hence the SBS. Based on the proceedings, further investigations are required.

Table 2: Comparison of shear bond strength between the experimental groups

	TF0C	TF3C	Test of significance
Shear Bond Strength	8.03± 1.56 ^{A}	5.13± 0.56^{B}	P<0.001*

P: probability
Different superscripted letters denote significant differences between studied groups by Post Hoc Tukey test.

Conclusion:

Within the limitations of this study, the incorporation of 3 WT.% of TiF4 within the primer of 3-step etch and rinse orthodontic adhesive was not recommended. Although it did not affect the degree of conversion, the bonding potency was greatly compromised.

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