

Physical and Chemical Mechanisms Involved in Adhesion of Orthodontic Bonding Composites: in Vitro Evaluations

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Physical and chemical mechanisms involved in adhesion of orthodontic bonding composites: in vitro evaluations

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'Declarations':

Ethics approval and consent to participate

All procedures performed in studies involving human participants were in accordance with the ethical standards of the University of Rome “Tor Vergata”, Rome, Italy, and with the 1964 Helsinki declaration and its later amendments. Human premolars, previously extracted for orthodontic reason, have been employed with the approval of the Ethics in Research Committee of the Centre of Health Sciences of the University of Rome “Tor Vergata”, Rome, Italy. Informed consent was obtained, at the time of extraction, from all individual participants included in the study, as part of routine documentation. Patients were informed that the extracted teeth could be used for research purposes, and written approval (by signature) was obtained from the patients at this point. Written informed consent was obtained from a parent or guardian for participants under 16 years old.

Consent for publication

Not applicable.

Availability of data and materials

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Competing interests

The authors declare that they have no competing interests.

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Abstract

Background

Bond strength of orthodontic composite is strongly influenced by molecular and structural mechanisms. Aim of this in vitro study was to compare the bond strength of light-cure orthodontic composites by measuring debonding forces and evaluating locations of bond failure. Investigations on chemical compositions clarified adhesive behaviors and abilities, exploring effects of ageing processes in this junction materials.

Methods

Sixteen human premolars were selected to realize specimen of enamel sections, randomly divided into three groups, which was assigned a different orthodontic adhesive system and then underwent to Shear Bond Strength test.

Twenty-seven human premolars were selected to produce samples, each of which was coupled at a metallic bracket, bonded to the enamel tooth surface with one of the three orthodontic adhesive systems, to quantify, at FE-SEM magnifications, after debonding, the residual material on enamel and bracket base surfaces.

Eight test discs of each orthodontic composite, were divided into three randomly groups to study accelerated aging effects in human saliva and sugary drink. Chemical compositions of composite materials were investigated, before and after ageing procedures, by Raman Spectroscopy analysis.

Conclusion

The orthodontic adhesive systems showed rather similar strength of adhesion to enamel. The breakage of adhesive-adherent bond occurs in TXT at enamel-adhesive interface while in Bisco and Leone composites at the adhesive-bracket interface. Accelerated in vitro aging demonstrated a good

physical-chemical stability for all orthodontic composites, Bisco only, was weakly contaminated with respect to the other two materials.

Keywords

Light-cure orthodontic composites, Shear Bond Strength, Field Emission Scanning Electron Microscope, Weight loss analysis and Raman Spectroscopy.

Background

Adhesion can be defined as the sum of the chemical and physical forces that represent the molecular attraction between materials in close contact. It expresses the resistance to separation forces. Adhesion phenomena are critical in many clinical applications of dental materials, including orthodontic bonding. The success of the adhesion is strictly linked to the characteristics of the interfacing surfaces and to the properties of the material used as bonding [1].

In orthodontics, polymeric adhesive resins are widely used as a dental bonding system to ensure an intimate and strong joint between the base of the bracket and the enamel surface.

The ideal adhesive system should have an optimal bond strength, which is able to resist both masticatory and orthodontic forces and should also allow for easy and safe detachment of the bracket, first of all avoiding damage to the enamel and then the persistence of residues in situ, which would require further mechanical removal steps, by means of drilling or air abrasion, which could in any case alter the roughness of the tooth surface [2].

Currently, in the scientific literature, it is not possible to find a uniformity of opinion on what is the optimal value of the bond strength between direct bracket and tooth enamel, in the clinical use.

I.R. Reynolds suggested a minimum value, i.e. valid for most of the clinical orthodontic needs, of the bond strength that orthodontic adhesives must have, it was estimated to be 6-8 MPa [3,4]. This value turned out to be higher than that of 5.1 MPa proposed a few years before by F. Miura [5].

J.L. Lopez established that a value between 3 and 5 MPa is more than enough to satisfy orthodontic requests while, T.W. Orthendahl proposed values in the order of 4 MPa [6,7].

Finally, W.A. Brantley and T. Eliades observed the existence of orthodontic adhesive systems whose shear bond strength can vary within an even greater range, that is, even between 8 and 30 MPa. The authors considered that the average force transmitted to a bracket during chewing can vary between 40 and 120 N and that its adhesion to the enamel surface requires a minimum force of 120 N (12.2 kg or 5.6 lb). Since that the most commonly used brackets have a hypothetical adhesion area of 16 mm² and then, by measuring the force necessary to obtain the bracket debonding, which turned out to be on average 120 N, they were able to calculate the minimum value of the adhesion strength, i.e. equal to 7.5 MP [8].

At the end of the treatment, the bond strength must be of such an extent as not to cause cracking or chipping (defects) on the surface of the dental enamel and prosthetic crowns [9]. For this reason, A.M. Compton reported that 7 MPa is the maximum value of adhesion to the enamel which would allow to avoid precisely the onset of such problems [10].

The best way to test a biomaterial is certainly its long-term clinical use, but also the in vitro measurement of detachment forces and adhesion plays an important role in the characterization of the adhesive potential of the orthodontic adhesive systems. In light-cured composites, the light-curing process does not always occur homogeneously throughout the material. The formation of radicals begins first on the surface exposed to sufficient light intensity. It is its fluidity that allows radicals to completely penetrate the entire structure of the material in order to carry out a complete radical cross-linking reaction [11,12].

The bond strength of orthodontic adhesive composite used for bracket bonding appear to be strongly influenced by both molecular and structural mechanisms.

Therefore, the aim of this in vitro study is to compare the bond strength of three different light-cure orthodontic adhesive resins by measuring the force of debonding and evaluating the location of bond failure. Subsequently, the chemical composition of the dispersed phase is investigated to explain how they affect the adhesive behaviour not only at the adhesive / enamel interface but at the adhesive / bracket base interface and how they are responsible for the ability of adhesion. To fully comprehend the material behaviour during its clinical use, we evaluate also the specimen properties after aging analysis in human saliva and sugary drink to understand if these effects can play a role in the alteration of the chemical structure of materials thus contributing in changing the adhesion properties of the resins.

Materials and Methods

Shear Bond Strength (SBS) test

Sixteen human premolars, previously extracted, with the approval of the Ethics in Research Committee of the Centre of Health Sciences of the University of Rome “Tor Vergata”, for orthodontic reasons, Italy, were selected, cleaned and stored in normal saline at 37 ° C. For each tooth, the crown was separated from the respective roots below the amelo-cement junction, subsequently the cusps were removed with a 0.3 mm thick diamond separator disc, thus obtaining a section of enamel only, as thick as possible, with two surfaces perfectly parallel to each other. Each of the enamel sections obtained was inserted in the middle of a hollow plastic cylinder, with its occlusal face facing upwards. At this point, self-curing acrylic resin was poured into the cylinder until it was completely filled and taking great care not to cover the enamel section inserted. Once the acrylic resin had hardened, the

sample was sawn and flattened to be flush with the tooth surface. The enamel disc was then finished first with a rubber cup and then with a silicon carbide polishing paper used, after softening with water. Each sample was placed in a mould to allow the correct compaction of the orthodontic resins. Each disc obtained was inserted horizontally inside a support with screws, which had a surmounted cylinder on its upper part that allowed the adhesive orthodontic resin to be perfectly in contact with the surface of the enamel, so as to create, precisely adhered to it, an orthodontic resin cylinder of a suitable diameter for testing with Ultra Tester Machine 91099/KB3 (Ultradent).

The sixteen disks obtained were randomly divided in three groups (G₁, G₂, G₃) and each group was randomly assigned a different adhesive system used for bracket bonding [Table 1].

	<i>Orthodontic adhesive system</i>	<i>Manufacturer</i>
TXT	Transbond XT™ Light Cure Adhesive is composed of a primer in bottle and a paste in syringe, 37% orthophosphoric acid (ETCH-37TM, BISCO) in add	3M Unitek, Monrovia, CA, USA
LEONE	Light-Cure Orthodontic Paste is composed of a paste in syringe (F3172-01), a primer in bottle (F3171-01) and an acid etch (F3143-01)	Leone spa, Sesto Fiorentino. FI, Italy
BISCO	Bisco Ortho Bracket Paste LC is composed of a paste in syringe, a one-step primer in bottle and an etching gel (ETCH-37TM, BISCO) in syringe	Bisco, Schaumburg, Illinois, USA

Tab.1: Light-cured orthodontic adhesive systems considered in the study

The occlusal surface was first cleaned with silicon carbide polishing paper and water and then dried with a jet of air. Etching gel was applied to the tooth surface for 30 seconds. After rinsing and air-drying, a uniform layer of primer was applied with a brush on the surface to be bonded, using circular movements, for 30 seconds. The primer was polymerized with the lamp for 30 seconds. The disk was inserted in the special support and the adhesive resin was brought into the cylindrical mould by a small spatula and compacted with a ball shutter. Finally, polymerization was carried out with a VALO curing light (Ultradent), according to the times indicated by the manufacturer, from top and side.

The disks, after 12 hours stored in normal saline at 37°C, were inserted, one at a time, vertically inside a metal support. The machine was equipped by a holder to locate the samples and a stab that applies

a shear stress force over the cylindrical sample until reaching bond rupture, obtaining the maximum tensile shear force.

Twelve measurements for each orthodontic adhesive system were obtained using a specific force for 60 seconds. At the end of the test it is possible to see on the display of the machine the peak of force to which the material has resisted before detaching from the enamel surface. The samples were used several times, each according to the amount of residual enamel after each test and after removing the residual material, up to a maximum of 4 times for a total of 36 measurements (12 per material).

Field Emission Scanning Electron Microscopy (FE-SEM) characterization

Twenty-seven human premolars, extracted with the approval of the Ethics in Research Committee of the Centre of Health Sciences of the University of Rome "Tor Vergata", for orthodontic reasons, were selected, cleaned, stored in normal physiological solution at 37 ° C and subsequently randomly divided into three groups (n = 9), each of which was coupled at one of the three orthodontic adhesive systems under examination.

The root of each premolar was inserted, until reaching the amelo-cement junction, inside a base prepared with Tenatex red wax (Kemdent), to better ensure the stability of the tooth during bonding manoeuvres.

The operative procedure involved the initial cleaning of the enamel surface with pumice and water, followed by drying with a jet of air.

Then, Ovation (Densply GAC International, Bohemia, NY, USA) stainless steel brackets equipped with 3-layer Supermesh base were tested. Each bracket was bonded, always by same operator, to the vestibular surface of the assigned tooth, respecting the orthodontic direct bonding protocol provided by the adhesive material manufacturer.

All the orthodontic adhesive systems have been light-activated as recommended by each producer, applying a light-curing unit (LCU) (LED Starlight lamp), whose power density was previously measured with a curing radiometer (Model 100, Demetron Research Corp. Serial n° 129540) and then set at 400 mW/cm², according to the pulse-delay light-curing methodology and placing the tip of the light unit at a minimum distance of 1.0 mm from the dental enamel surface.

All the specimens obtained were stored in normal saline at 37°C for 24 hours, in order to facilitate the polymerization and the hydration of both the material and the tooth. The same operator also carried out the detachment of the brackets from the surfaces of the tooth enamel, carefully inserting the working part of a stainless steel ligature pliers (P 1919-00, Leone spa), in the interface between the metal base of the bracket and the orthodontic adhesive resin, making sure to reproduce always the same twisting motion. Morphological investigations of the brackets and premolars on which they were previously attached, were carried out through image acquisitions, from 5000 to 60000x, obtained by Leo Supra 35 FE-SEM Field Emission Scanning Electron Microscope (Carl Zeiss, Germany).

Weight loss analysis

With a thermo-formed polyurethane mould, previously made that reproduced inside 3 negative discs (10.0 mm in diameter and 4.0 mm thick), specimens of each material to be tested were obtained. Each disc has been completely filled with one of the three orthodontic resins considered in the study and before the polymerization process, a transparent strip (Hawe Neos Dental, Bioggio, Switzerland) has been placed and pressed to create the smoother and more uniform surface as possible. Glass plates were placed on the top and bottom of the mould to provide flat surfaces.

A light-curing unit (LCU) (LED Starlight lamp) has been used, whose power density was previously measured with a curing radiometer (Model 100, Demetron Research Corp. Serial n° 129540) and then set at 400 mW/cm².

The orthodontic composite resins have been light-activated as recommended by each manufacturer, applying the LCU at the top and bottom surfaces, where the light tip was placed in contact with the glass plate at a distance of 1.0 mm from the specimens.

Eight test discs (10.0 mm in diameter and 4.0 mm in thick), made of each orthodontic resin in exam, have been obtained for a total of 24 specimens. Samples were divided into 3 randomly groups and stored in distilled water at 37 ° C until they have been used for the study of the accelerated aging effects to verify the chemical stability of the materials. For this purpose, they were immersed for predetermined times of 1, 7, 14, 21 and 28 days in two different solutions able to simulate the hostile environment of the oral cavity: sugary drink and human saliva [Tab. 2].

Testing Solutions	Description
Sugary drink	5 ml of Coke ®
Saliva	5 ml human saliva collected according to standard protocols from healthy male volunteers, no smokers or drinkers, aged between 25 and 40 years

Tab. 2: Ageing solutions considered in the study.

Each sample was placed into an Eppendorf test tube, immersed in its corresponding ageing solution at fixed temperature of 37 ° C, at established time intervals, until one month of storage have been got. Different samples for each material have been tested and weighted 5 times with a precision balance (Mettler Toledo) after each time interval. For each weighing, the samples were pulled out from the tube, rinsed in deionized water for 5 minutes and dried in a nitrogen flux. Furthermore, before the measurement phase, they were stored 3 hours in a glow box at 30 ° C and 30 % of humidity, to promote the complete water evaporation on the composite materials. During the handling of the samples, a Teflon tweezer was used which ensured to avoid any accidental damage or any change to

the resins surfaces. In order to compare samples different in size and weight the percentage weight relative variation has been calculated. Finally, the average and the standard deviation of the relative percentage weight variations have been got.

Raman Spectroscopy analysis

A Thermo Scientific DXR Raman Microscope has been used to investigate the chemical composition of the materials before and after the ageing procedure in saliva and sugary drink. A 532 laser source powered at 10 mW with an exposure time of $t = 1$ s for 200 accumulations was employed. The spectra were acquired in the range $300\text{-}3300\text{ cm}^{-1}$ with a 50x objective and a 5th order polynomial correction was used to correct for fluorescence contribution.

Statistical analysis

Data were analysed using Kruskal-Wallis and Mann-Whitney-U tests; Bonferroni Scheffe, and Sidak multiple comparison tests were used, p values were computed and compared with statistical significance at the $p=0.05$ level. The data were analysed with the statistical software STATA (STATA Statistical Software release 12.1; Stata Corporation, College Station, TX).

Results

SBS test results

The collected data have been summarized in **table 3**, which indicates, for each orthodontic adhesive system under examination, the SBS values, expressed in newton (N).

Measurement	TXT SBS (N)	LEONE SBS (N)	BISCO SBS (N)
n. 1	20.4	7.4	4.2
n. 2	19.2	5.6	13.3
n.3	10.1	17.2	12.5
n. 4	18.9	9.9	16.1
n. 5	17.6	9.9	12.3
n. 6	9.5	9.4	14.9
n. 7	17	11	10.2
n. 8	6.5	6.1	8.6
n. 9	17.6	8.8	23.2
n. 10	13.9	15.3	14
n. 11	5.9	10.4	14
n. 12	11.3	7.3	10.6

Tab. 3: Shear bond strength test values on the three orthodontic adhesive system examined.

Statistical analysis of the values obtained from the 12 measurements performed on each material has been executed and the average, standard deviation (DS), minimum (Min) and maximum (Max) values of SBS, expressed in megapascal (MPa), have been shown [Tab. 4].

Orthodontic adhesive system	Average SBS (MPa)	DS SBS (MPa)	Min SBS (MPa)	Max SBS (MPa)
TXT	4.45	1.32	1.88	6.49
Leone	3.14	1.51	1.78	5.47
Bisco	4.08	1.73	1.34	7.38

Tab. 4: Average, standard deviation (DS), minimum (Min) and maximum (Max) values of SBS, expressed in MPa.

Specifically, the values in N describe the SBS considering the surface of the retentive base, while the values in MPa, obtained by dividing the values in newton by the base areas, exclude the influence of the millimetre adhesion of the base and strictly reflect the effectiveness of the retention mechanism.

The average SBS obtained is equal to 4.45 MPa for TXT, to 3.14 MPa for Leone and to 4.08 MPa for Bisco (Fig.1).

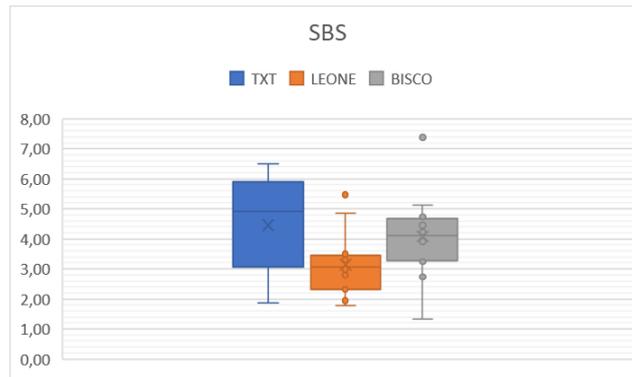


Figure 1: Scatter plot of the SBS values of the three orthodontic adhesive systems, expressed in MPa.

SEM characterization results

The most significant images obtained at SEM were found to be those relating to the residual adhesive material on the base of the brackets and are illustrated in figure 2 (a, b, c).

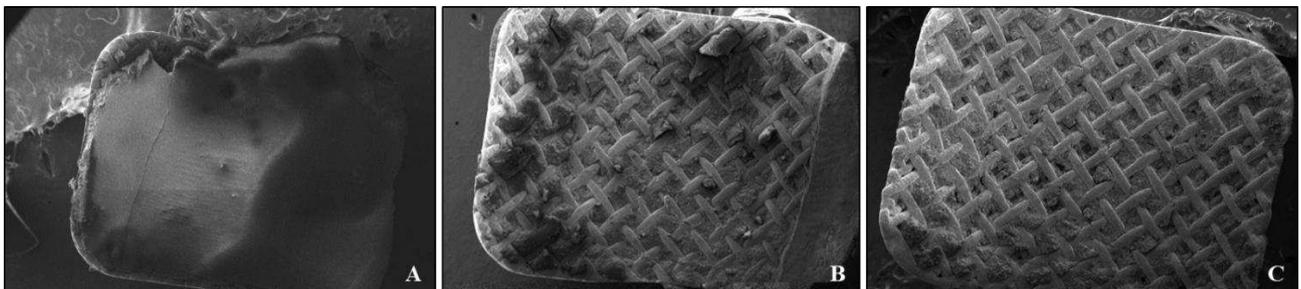


Figure 2: SEM magnifications relating to the material residues of the three orthodontic adhesive systems that remain on the base of the brackets after debonding, in particular it is observed in a) TXT (51x), b) Leone (54x) and finally c) Bisco (59x).

Weight loss analysis results

The different adhesive materials have been stored for one month in sugary drink or saliva and chemical stability and weight losses have been determined.

After weightings, none of the specimens have shown significant weight loss values, thus proving a good material stability. As expected, it was registered a slight increase in their weight for all the materials due to the absorption of organic residues in the vial during the samples storage.

An interesting argue is related to the saturation of this weight change depending by the type of storing liquid and by the time. In figure 3, it is possible to observe the relative weight shift for the samples stored in saliva and sugary drink for the different materials.

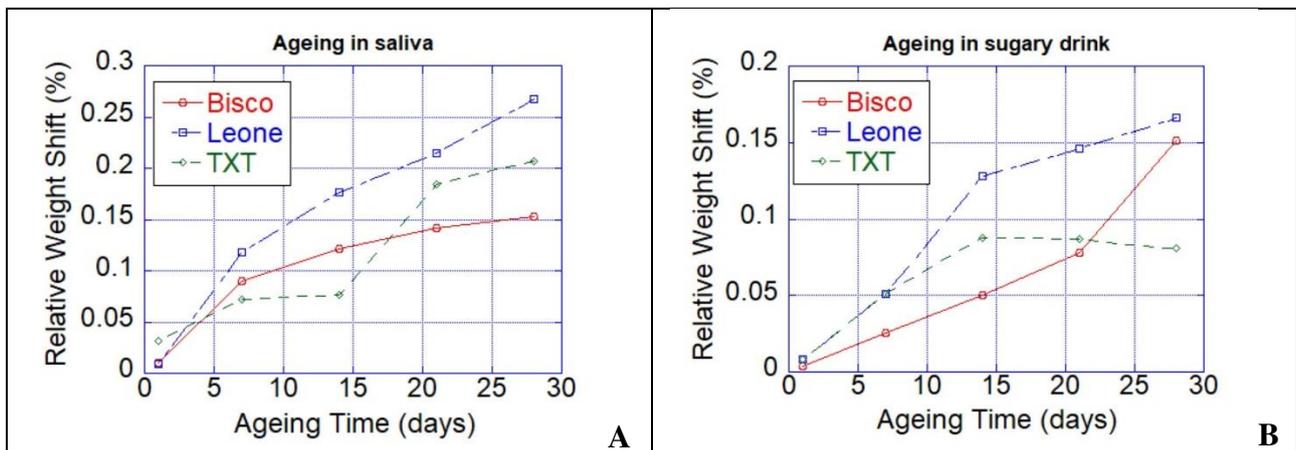


Fig.3: a) The relative weight shifts for the three orthodontic adhesive systems samples stored in human saliva; b) the relative weight shifts for the three orthodontic adhesive systems samples stored in sugary drink.

In saliva ageing (Fig.3a), Bisco and Leone, show a very similar trend with a monotone increasing, while in TXT is possible to observe a plateau up to two weeks and a following increasing of the weight. This behaviour can be related to a change in the absorption of TXT during the ageing. In any case, after one month Bisco exhibits the most stable performance.

For ageing in sugary drink (Fig.3b), a similar trend for Bisco and Leone is shown, while TXT reveals a weight saturation after fifteen days, thus demonstrating a different type on interaction with the testing solution.

Raman Spectroscopy analysis results

In Figure 4 the comparison of the Raman spectra of the composition of the three orthodontic adhesive resins has been illustrated.

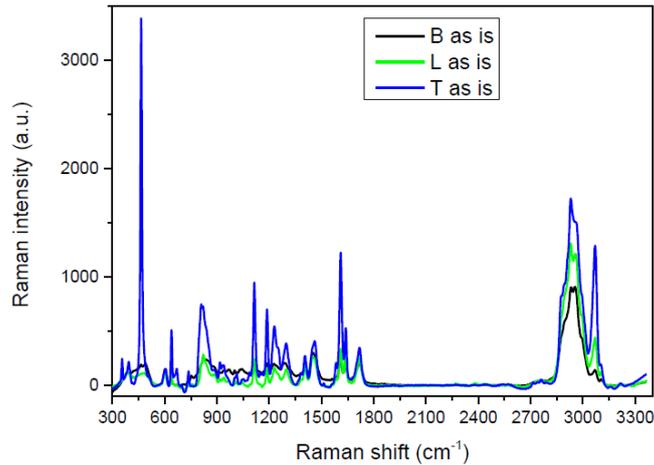


Fig. 4: Comparison among the Raman spectra of the three investigated materials.

In Figure 5 the comparison between the Raman spectra collected on Bisco (Fig.5a), Leone (Fig.5b) and TXT (Fig.5c) before and after ageing in saliva and sugary drink has been presented.

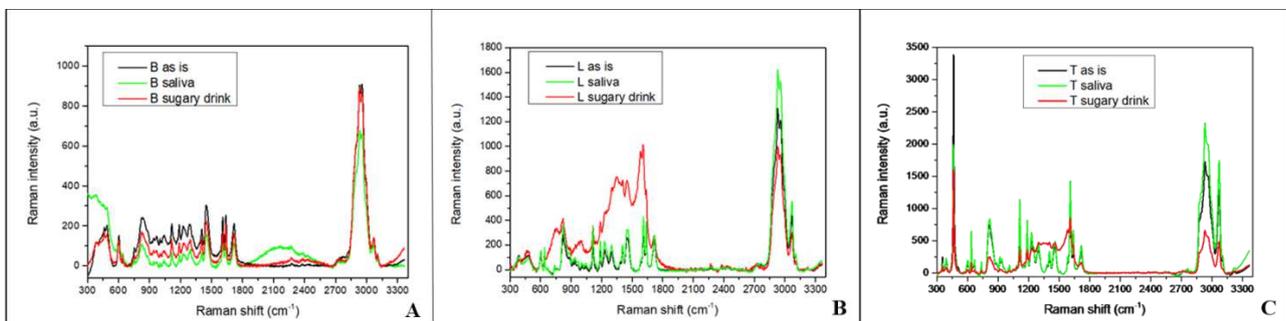


Fig. 5: a) Comparison between the Raman spectra collected on Bisco before and after ageing in saliva and sugary drink; b) comparison between the Raman spectra collected on Leone before and after ageing in saliva and sugary drink; c) comparison between the Raman spectra collected on Transbond XT before and after ageing in saliva and sugary drink.

Discussion

The bond strength, that the orthodontic adhesive system is able to generate, influences the outcome of the bracket bonding to the surface of the dental enamel. This is essential for the success of the treatment since, from a clinical point of view, phenomena of accidental debonding could be the cause

of damage to the dental enamel, increase the number of appointments necessary and / or extend both the operating times and those necessary to complete the orthodontic therapy [13]. Specifically, the incidence of the detachment phenomena was described both at the enamel - adhesive system interface and at the adhesive system - bracket base interface. It has been observed that these phenomena depend on the value of the bond strength of the orthodontic adhesive, but are influenced also by high mechanical stresses that occur during the orthodontic therapy, or as a result of a decrease in the bond strength at the interface, as occurs when using brackets in polymeric material [8].

F.L. Romano evaluated in vivo the failure rate of adhesion of metal brackets to the tooth enamel surface of both arches with the TXT adhesive system. It was found to be equal to 1.57%, i.e. only 3 brackets out of 190 in a 6-month period underwent accidental debonding [14]. These data are indicative of why this material is, to date, the most widely used adhesive system, as a control, both in clinical and laboratory studies and why TXT has been considered in our in vitro study, despite the abundant scientific literature already provided on it [15-22].

In order to investigate the orthodontic bond strength, the majority of researchers used the strength of the "shear" bond rather than tension or torsion, as the former was found to be the most reproducible. As regards the values of the orthodontic bond strength, reported in the literature, it is actually observed that they can vary considerably also depending on the specimen preparation specifications and the tests conditions [23-34].

C. Sturdevant has shown that, depending on the material of which the bracket is made of, the value of the bond strength of the adhesive system, obtained following mechanical tests of resistance to shear strengths, can vary between 17 and 24 MPa [35]. In this regard, T.R. Katona has conducted extensive researches demonstrating that this simple approach of measuring the strength of adhesion, can provide often incorrect results [36]. Indeed, the stress produced on the bracket and on the enamel is not homogeneous, but it is concentrated, generating a local stress greater than that created between the applied force and the interface. The result is an underestimation of the local stress, that causes the

failure of the adhesive bond, caused by micro-cracks propagation through the adhesive itself, which is more fragile and, probably, also through one or both interfaces [37,38].

N. Fox hypothesized an existing relationship between the force application site and the surface of the base of the bracket, noting that a variability in the arrangement of this site and the relative position of the constituents of the adhesion system (enamel-adhesive-bracket), is able to determine substantial differences in the measurement of that force which is responsible for the failure of the adhesive bond [39].

Results of tests with tensile or shear strength can determine a coefficient of variation or relative standard deviation [(standard deviation / mean) x 100%] ranging from 20% to 30%. Typically, tensile force tests produce a lower coefficient of variation than the more common shear strength analysis.⁸ Currently, a standard for assessing the bond strength to tooth enamel, or to other surfaces, of orthodontic adhesive systems is not clearly reported in the literature however, it is established that some factors must be considered, such as: the method of load application, the penetration speed, or crosshead speed, of the machinery used for the test (usually equal to 0.5 mm / min); the design of the bracket and the statistical analysis of the data [8].

By comparing the data obtained in our study, it is possible to highlight how all the adhesive systems under examination provide average SBS values in the range of 3 - 4.5 MPa. As shown in Tab. 4, TXT, with an average SBS value equal to 4.45 MPa, was found to be the adhesive material with the greatest adhesion strength, on the contrary Leone proved to have the lowest average SBS values, in fact equal to 3.14 MPa. The Bisco, on the other hand, showed intermediate SBS average values, equal to 4.08 MPa, but with larger variations from the lowest (1.34 MPa) to the highest (7.38 MPa) measurement among all the samples examined [Tab .4].

However, it is important to underline that all the data collected by us are far from the parameters suggested by I.R. Reynolds as clinically acceptable levels of adhesion strength, i.e. 6-8 MPa [40]. Various studies have suggested bond strengths between 2.8 and 10 MPa to be clinically adequate

[4,5]. The same comparison, concerning the TXT and the SBS parameters present in the literature, shows an important difference in the results obtained in our study [Tab. 4] [41]. This significant discrepancy in the results is most likely explained by the fact that our study was purposely conducted without the use of brackets since, from the literature it is clear that the differing geometries of the bases of the bracket are able to greatly influence the strength of adhesion of orthodontic adhesive systems [41]. However, the purpose of our study was to compare the adhesion capacity of the three orthodontic adhesive systems in question and for this reason we chose to isolate the "bracket base" variable, focusing attention on the real resistance that the resin alone offers shear forces.

The objective of many studies in the literature is to demonstrate, following the detachment of the bracket, at what level the breaking of the bond of the bracket to the tooth surface occurs. When testing for adhesive bond failure, three situations may arise: bond failure at the base bracket / adhesive interface, at the adhesive / enamel interface, or cohesive failure [42]. The literature shows that the fracture gap can be localized, to the same extent, both at the enamel / adhesive interface, and at the bracket / adhesive interface [43-49].

The orthodontic adhesive resin however remaining on the enamel surface, after the bracket debonding, is necessarily mechanically removed from the tooth surface by milling and this entails the risk of accidentally removing even the most superficial layer of the dental tissue [25].

According to S. Elekdag-Turk, the prevalence of adhesive bond failure at the bracket / resin interface becomes a protective phenomenon for the enamel, precisely because, at the moment of detachment, this structure remains intact, preventing damage such as the loss of superficial tissue fragments therefore, cleaning the dental surface from the residual adhesive resin is perhaps less risky than the damage induced by bracket debonding [50]. Regarding the breaking of the bond at the enamel / adhesive interface, according to T. De Melo, the anatomy, the curvature and the design of the base of the bracket, may be the factors responsible for the greater strength of the bonding at bracket / adhesive interface and is just this condition to favour the major preservation of the dental enamel, because only

a thin layer of residual material remains to be removed by milling [51].

It is also true that the removal of a bracket attached to the tooth through a high SBS adhesive system can increase the incidence of fractures or micro-cracks affecting the enamel, because of the increased effort have to be spend to remove the retained adhesive resin from the enamel surface [25]. Actually, what is considered as desirable in the debonding milling manoeuvres is to remove the bracket without damaging the enamel surface. It was also quantified that even the safest debonding manoeuvres were found to be responsible for the loss of about 10-20 μm of surface enamel [8].

The FE-SEM analysis of the bracket bases coupled at one of the three orthodontic adhesive systems under examination, have identified the adhesive interface in which the fracture and detachment of the bracket most commonly occurred.

Fig. 2 illustrates the most frequently observed behaviour for each orthodontic adhesive system and significant differences between TXT and the other two resins can be noted. The images show that the breakage of the adhesive-adherent bond occurs, for the TXT, at the enamel-adhesive interface, given the large amount of resin residual on the base of the bracket (Fig.2a). Highly filled resin composites have been observed to bond to mechanically retained metal brackets better than lightly filled composites [52].

The situation in Bisco (Fig.2c) appears different: given the scarce remaining adhesive material on the retina of the bracket base, is evident that the detachment at the adhesive-attachment interface occurred. Finally, the Leone (Fig.2b), shows a more similar performance to Bisco adhesive resin.

As regards TXT, which once again is the material repeatedly subjected to tests, some of the studies in the literature presented showed a tendency for the adhesive bond to break mainly at level of the enamel-adhesive interface, rather than the adhesive-bracket one [25,27,28,30,53]. This latter evidence is precisely in accordance with the results obtained in our in vitro study.

It has been reported that, enhancement of bond strength may compromise safe bonding, in fact, the detachment of a large part of TXT from the dental surface could expose, even if in a minimal percentage (1-30%), the enamel to damages such as micro-fractures or loss of superficial hard tissue. Bisco and Leone, which have instead shown a greater tendency to remain adherent to the enamel surface, have to be mechanically removed from the tooth by mechanical milling, taking care not to damage the dental enamel. It has been observed that, the resin tags that remain on the enamel surface, for a long time after the bracket debonding, can change colour as well as constitute sites for bacterial adherence [54].

The FE-SEM images however show a good performance of the Ovation bracket in most of the samples examined. The 3-layer Supermesh net seems to be able to retain a good amount of adhesive resin between its meshes.

The investigation of aging in saliva and in sugary drink has been performed with the intent to establish whether the recorded dimensional variations, even if minimal, were still capable of altering the physical-chemical properties of the surface of the three orthodontic composite resins under examination. Data on weight changes in saliva showed a small weight increase in the order of 10^{-4} g, which is however considered not significant in terms of the chemical stability of the examined materials. These minimal modifications in weight values can be expected and caused by the surface adsorption phenomenon of organic saliva residues, as proteins and mucus. Overall, this enhancement in weighting resulted not uniform in the trends belonged to each orthodontic adhesive resins, although it showed a tendency to stabilize over time. In figure 3a Bisco and Leone resins demonstrate a very analogous pattern remaining stable in the first period of in saliva immersion and then increase, about 2×10^{-4} g / 6×10^{-4} g in weight, at regular intervals of 5 and 15 days. The interaction of TXT resin with human saliva showed a different model trend (Fig. 3a), by which, during the first 5 days of immersion, it tended to arise its weight of 4×10^{-4} g in and then remain almost steady over the next 10 days. This second monotone phase then changes rapidly over the last 15 days, with a sudden increase in weight,

in the order of 1×10^{-3} g. Even in this case, the alteration in weight is probably due to absorption phenomena of various organic substances contained in saliva as the result of reversible interactions, as it does not imply any type of irreversible chemical reaction as can be seen also by the unchanged colour of the sample surface. The investigation of ageing in saliva, it has been followed that the minimal dimensional variation recorded in the tested orthodontic resins does not able to alter physical-chemical properties in the surface.

As showed in figure 3b, which summarizes the weight measurements of the samples over 30 days, even in this case the trends of Bisco and Leone resins can be overlapping. Both materials have a total weight increase of 1×10^{-3} g, which is regular in all the 30 days. Every 5-10 days, there was an increase of 2×10^{-4} g in both samples, with a starting tendency to a stabilization, as indicated by the Weight/Aging curve. The TXT resin, instead, interacted differently with the sugary drink: during the first 15 days of immersion, weight increase has been recorded at regular intervals of 2×10^{-3} g every 7 days. Subsequently, it has been characterized by an enhancement of 6×10^{-3} g over the last 15 days occurred. About weight changes for samples stored in sugary drink, it is showed a similar behaviour than which one recorded in saliva: a different interaction of the TXT is recorded both in saliva than in the sugary drink respect Bisco and Leone adhesive resins.

Overall, the weight changes of the three materials are again considered almost negligible and no materials losses can be evaluated in all the tests; therefore, all the orthodontic resins have shown to be resistant to the acidic and corrosive components of the sugary drink used in the experiment.

Raman Spectroscopy was a powerful tool, it has been used to investigate on the chemical composition of the three orthodontic adhesive resins, especially on the nature of their dispersed phase and, to establish the effects that occur following phenomena of aging in saliva and sugary drink, precisely at the level of the surface. Therefore, the purpose of this analysis has been twofold: a simple compositional comparison of the three adhesive resins (Fig.3b) and an evaluation of the quality of the three materials (Fig. 4) in response to an exposure, until one month, in two different aging solutions

to likely correlate these findings to possible changes in mechanical behaviour of the materials. On the basis of the chemical bonds reproduced in the Raman spectra it has been established that the type of filler prevalent in the dispersed phase of each resin is quartz. Strong affinities between the spectra of Bisco and Leone have been revealed, while the TXT exhibited a peculiar peak at 450 cm^{-1} associated to the higher percentage of quartz in its chemical composition (Fig. 4), as also reported by its safety data sheet. This last data allowed to justify both the diversity in the adhesive behaviour of the TXT resin and the similarities of performances demonstrated by the Bisco and Leone resins and finally, to justify the greater chemical affinity, or cohesion force which, following debonding, the TXT showed (fig. 2a) with the metal interface represented by the base of the Ovation bracket, compared to the other two orthodontic resins tested (Fig. 2b, c). It is known that a higher content in fused quartz fillers, involves the attainment of high compressive strength and stiffness, the abrasion resistance and, the reduction the thermal dimensional change of the resin to a value matching that of tooth structure, effectively increasing adhesion to both the interfaces: adhesive / enamel and adhesive / base bracket. The surface profile and microstructure of the orthodontic composites are subjected to changes arising from degradation and wear processes in service; through Raman analysis it was observed that, the ageing in saliva usually produces the appearance of new broad bands in the region ranging from 900 to 2500 cm^{-1} ascribed to a possible fluorescence signal coming from the adsorption of organic residues [55]. Here, all the three materials demonstrate exhibit a good behaviour showing small changes in the spectra without the presence of pronounced new bands, thus indicating the absence of relevant and permanent surface effects (Fig. 5).

In particular, for ageing in saliva, Leone and TXT do not show spectral variations, while Bisco presents a small intensity increase due to the presence of organic residues in the region between 1800 and 2700 cm^{-1} . Conversely, in case of ageing in sugary drink, Bisco does not reveal any variation in the spectrum while Leone and TXT show new contributions and a broad spectral convolution in the region $1200\text{-}1800\text{ cm}^{-1}$ due to surface alteration (Fig. 5a and 5b). Therefore, Raman analysis on samples stored in saliva and sugary drink highlighted that Bisco is weakly contaminated with respect

to the other two materials, and we speculate that this could be associated to peculiar moisture resistance properties.

Conclusions

Following the tests on the SBS, carried out on natural teeth and without the aid of brackets, a rather similar strength of adhesion to the enamel was recorded, clinically adequate and acceptable for the debonding manoeuvres, in all the orthodontic adhesive systems in exam. The greater SBS to dental enamel demonstrated by TXT, could increase the probability of iatrogenic lesions of the hard tissues of the tooth occurring during the manoeuvre of bracket debonding. On the other hand, the greater affinity for chemical bonding, or cohesion, of TXT for the metal surface of the base of the bracket, would guarantee the orthodontist better holding performance of the bracket, during the active phases of the treatment. FE-SEM bracket bases images show that, the breakage of the adhesive-adherent bond occurs for the TXT at the enamel-adhesive interface and al conversely, for Bisco and Leone, it occurs mainly at the adhesive-bracket interface.

At the investigation of aging in human saliva, it has been followed that the minimal dimensional variation recorded in the tested orthodontic resins does not able to alter physical-chemical properties in their surface and this also testifies to a good biocompatibility. Furthermore, all the orthodontic resins have also shown to be resistant to the acidic and corrosive components of the sugary drink used in the experiment. Finally, Raman analysis on samples stored in saliva and sugary drink highlighted that no significant chemical changes can be observed on the resins, thus do not altering the mechanical properties of the materials. In particular, after one month of aging, Bisco is weakly contaminated with respect to the other two materials, and we speculate that this could be associated to peculiar moisture resistance properties.

Abbreviations

FE-SEM: Field Emission Scanning Electron Microscopes

TXT: Transbond XT™ Light Cure Adhesive

Bisco: Light-Cure Orthodontic Paste

Leone: Bisco Ortho Bracket Paste LC

i.e.: *id est*

MPa: Megapascal

N: Newton

kg: kilogram

lb: libra

mm: Millimeter

°C: Degree Celsius

SBS: Shear Bond Strength

G: Group

Tab.: Table

n: number

MW: Megawatt

cm: centimetre

LCU: Light-curing unit

ml: Milliliter

%: percent

t: time

s: second

DS: Standard Deviation

Min: minimum

Max: maximum

Fig.: Figure

a.u.: arbitrary unit

min: minute

μm: micron

g: gram

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Figures



Figure 1

Scatter plot of the SBS values of the three orthodontic adhesive systems, expressed in MPa.

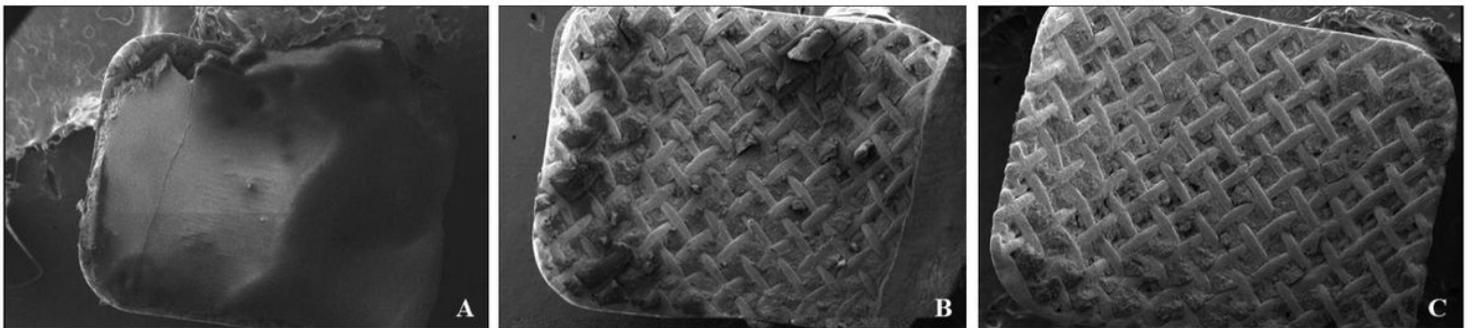


Figure 2

SEM magnifications relating to the material residues of the three orthodontic adhesive systems that remain on the base of the brackets after debonding, in particular it is observed in a) TXT (51x), b) Leone (54x) and finally c) Bisco (59x).

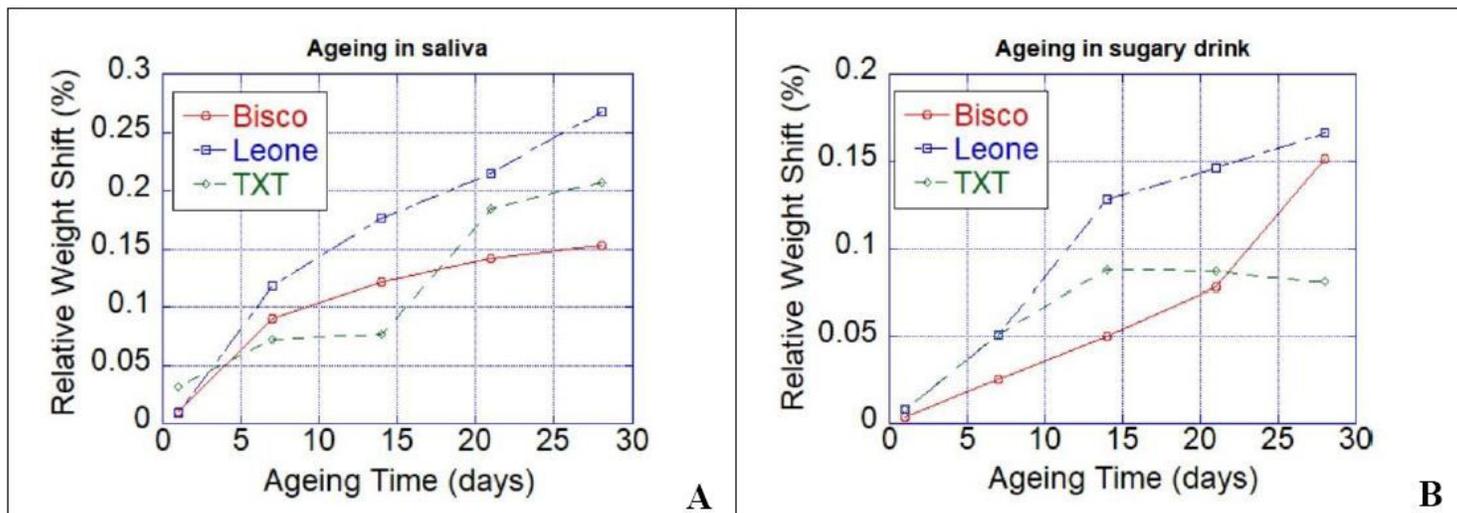


Figure 3

- a) The relative weight shifts for the three orthodontic adhesive systems samples stored in human saliva;
 b) the relative weight shifts for the three orthodontic adhesive systems samples stored in sugary drink.

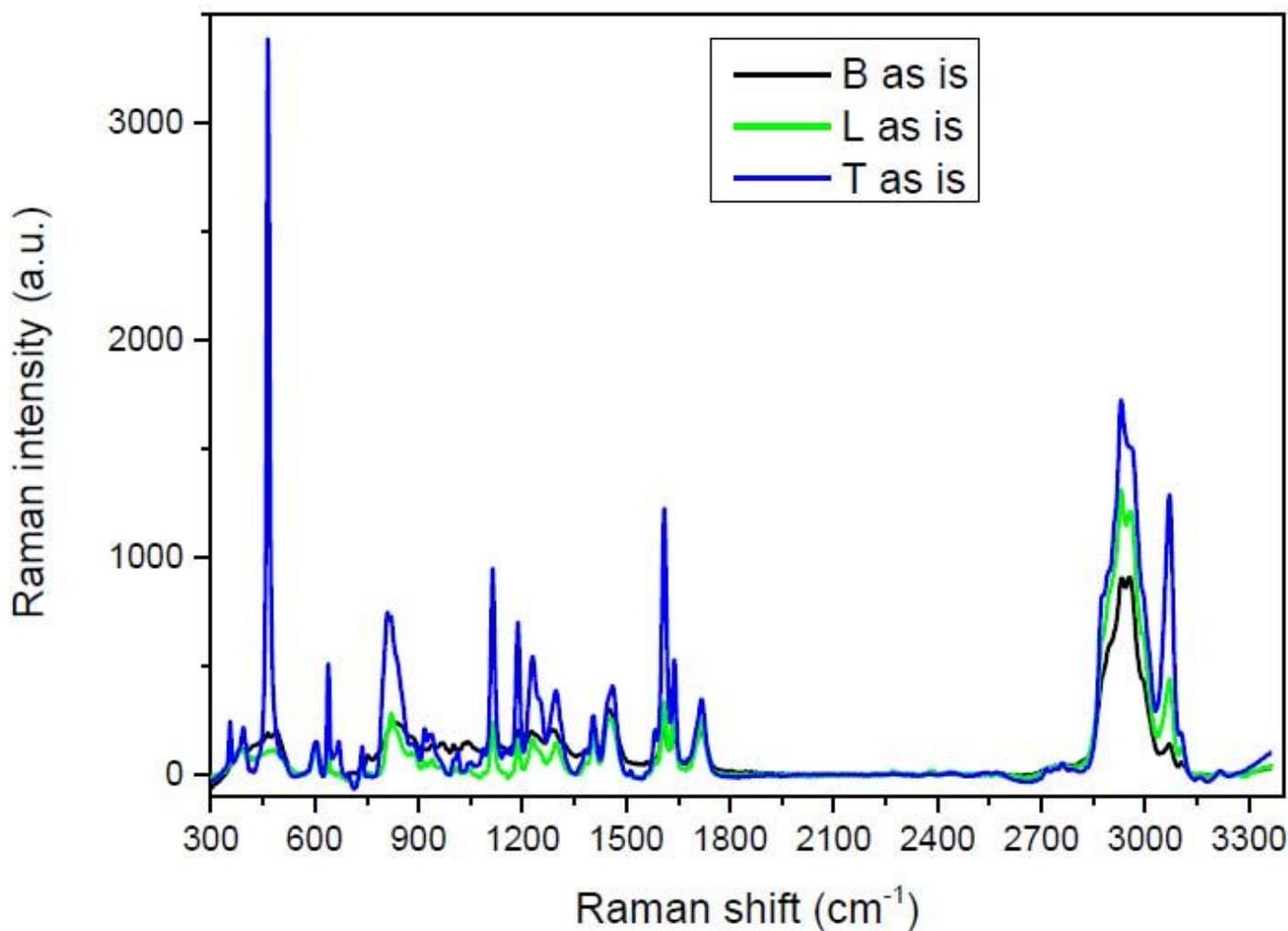


Figure 4

Comparison among the Raman spectra of the three investigated materials.

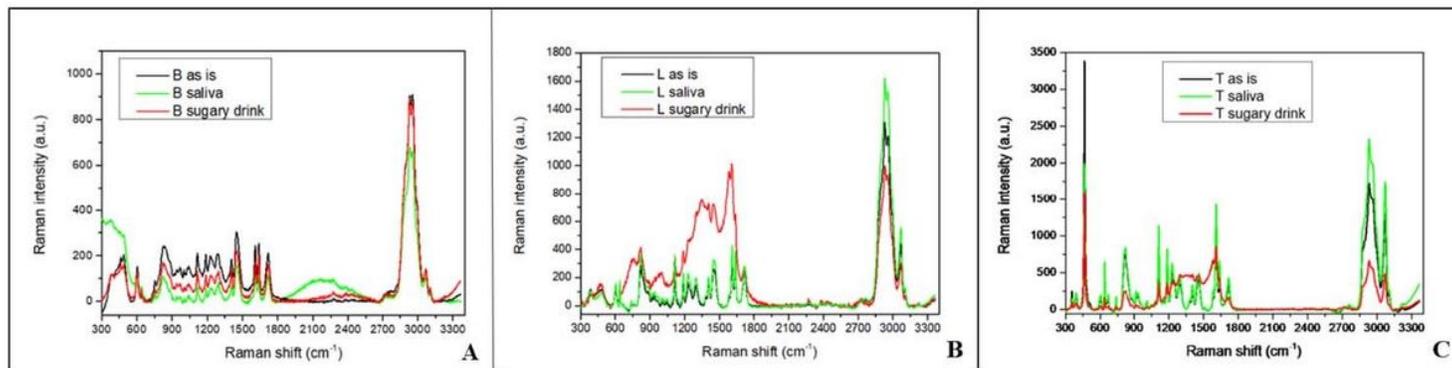


Figure 5

a) Comparison between the Raman spectra collected on Bisco before and after ageing in saliva and sugary drink; b) comparison between the Raman spectra collected on Leone before and after ageing in saliva and sugary drink; c) comparison between the Raman spectra collected on Transbond XT before and after ageing in saliva and sugary drink.