Assessment of degree of conversion of polymer-based orthodontic adhesive resins

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Abstract: Degree of conversion (*DC*) of selected currently used orthodontic adhesive materials, after their polymerization with dental curing light, were investigated in laboratory conditions. Samples of four orthodontic adhesives (Contec LC, Transbond XT, Transbond Plus and Resilience) were prepared in Teflon matrices and then formed into the thickness of 1 mm. After the baseline measurement of cross-linking of the materials with Fourier transform infrared spectroscopy (FT-IR) the samples were cured with treatment light, and then 1 and 24 h after the completion of the photopolymerization process, the values of *DC* were evaluated. The results were analyzed using the Statistica 8.0 software package. In the process of hypothesis testing the level of significance was assumed at $\alpha = 0.05$. The statistical analysis yielded significant differences between *DC* values of orthodontic adhesive systems. It was stated that *DC* values of the visible light-cured orthodontic adhesives assessed in the present study depend of the type of the composite material. A significant increase in *DC* occurring with extended period of observation proves that polymerization of dental adhesive materials is a long-term process.

Keywords: orthodontic adhesives, photopolymerization, degree of conversion, statistical analysis.

Ocena stopnia konwersji ortodontycznych żywic adhezyjnych na bazie polimerów

Streszczenie: Przeprowadzono w warunkach laboratoryjnych badania stopnia konwersji (*DC*) wybranych, stosowanych współcześnie ortodontycznych materiałów adhezyjnych po ich polimeryzacji pod wpływem światła widzialnego lampy stomatologicznej. Próbki czterech klejów ortodontycznych (Contec LC, Transbond XT, Transbond Plus i Resilience) przygotowano w teflonowych matrycach, a następnie uformowano do grubości 1 mm. Po wykonaniu za pomocą spektroskopii w podczerwieni z transformacją Fouriera (FT-IR) pomiaru początkowego usieciowania próbek, poddawano je działaniu lampy zabiegowej, a następnie po 1 i 24 h od zakończenia procesu fotopolimeryzacji wyznaczano wartości *DC*. Uzyskane wyniki analizowano korzystając z pakietu statystycznego Statistica 8.0. W procesie testowania hipotez statystycznych przyjęto poziom istotności $\alpha = 0,05$. Analiza statystyczna wykazała istotne różnice pomiędzy wartościami *DC* badanych klejów ortodontycznych wyznaczonymi po upływie 1 i 24 h oraz istotny wzrost *DC* podczas 24-godzinnego przechowywania ocenianych próbek. Stwierdzono, że wartość *DC* ocenianych układów adhezyjnych polimeryzowanych światłem widzialnym zależy od rodzaju materiału. Istotny wzrost wartości *DC* zachodzący wraz z wydłużeniem czasu obserwacji może świadczyć o tym, że polimeryzacja stomatologicznych materiałów adhezyjnych jest procesem długotrwałym.

Słowa kluczowe: ortodontyczne materiały adhezyjne, fotopolimeryzacja, stopień konwersji, analiza statystyczna.

The introduction of orthodontic adhesives based on composite materials into clinical practice, enabling attachment of orthodontic brackets directly to enamel's surface, was undoubtedly one of the milestones in the development of malocclusion treatment with fixed appliances. The use of adhesive techniques has reduced the need for orthodontic rings, which had a significant impact on the precision and comfort of doctor's work, as well as improved the comfort, safety and aesthetics of the use of fixed thin archwire appliances by the patient.

Since the 1970s, when adhesive materials based on composite resins came into use [1], dental composites have been subjected to continuous modifications aimed

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at improving their physical properties and chemical stability [2].

Orthodontic composite materials, as well as those used in conservative dentistry or prosthetics, consist of an organic matrix, inorganic fillers and additional components such as linking agents, initiators, activators, stabilizers, or dyes [3].

The organic matrix of orthodontic adhesives is made of so-called basic monomers or oligomers which are methacrylic acid derivatives and so-called "auxiliary" monomers with smaller molecules [3–5]. Basic and auxiliary monomers most commonly used for production of dental adhesive materials are listed in Table 1. The polymerization reaction makes them join into chains and networks, which manifests itself clinically with hardening of initially flexible or semi-liquid adhesive material, fastening elements of the orthodontic appliance to the tooth enamel. The type of used monomers, their quantitative composition, and the course and conditions of the polymerization process affect the polymer properties, *i.e.*, the chain lengths, the degree of branching, the number of crosslinks, and spatial structure. The chemical structure of the polymers in the organic matrix of the composite material determines the material's physical and chemical properties, that is, hardness, modulus of elasticity, the size of polymerization shrinkage, thermal expansion, solubility, or the degree of sorption of water [3, 5, 6]. The derivatives of the above mentioned features of bonding materials include: bond strength of orthodontic attachments to tooth surfaces, chemical stability of adhesive resins in the oral environment, aesthetics, safety of use, and biocompatibility of the material [7–10]. Pure polymer matrix would not fulfill clinical requirements for orthodontic adhesive materials such as resistance to physical factors, plasticity, and color, hence it was necessary to improve its properties by supplementing the material composition with inorganic fillers among which the most commonly used are glass, silica, or ceramics [3, 11].

The necessity of precise positioning of elements of fixed appliances on the tooth surface as well as work in a limited operating field exposed to moisture, also forced changes in the ways of initiation and propagation of the polymerization process. Originally used materials, polymerized solely through a chemical reaction initiated by an addition of a catalyst, have been largely replaced by light-cured materials, in which the creation process of the polymer spatial network is initiated by visible light. This allows precise control of the initiation of the material's polymerization reaction in clinical conditions, extending the time required for accurate positioning of orthodontic brackets and reducing the curing time of the adhesive [1–3].

One of the most frequently assessed parameters influencing chemical stability and physical properties of dental composites is the degree of conversion (*DC*) of their polymer matrix [3, 4, 7, 8, 11–14], specifying the percentage of conversion of double bonds of monomer or oligomer molecules into single intermolecular bonds. This mechanism is the essence of the polymerization reaction and leads to the formation of macromolecules of different length and structure [15, 16].

Numerous studies indicate that the crosslinking process of the matrix of composite materials is never complete, which is associated with the presence of residual monomer particles in the polymerized material, the presence of low-weight molecules, or molecules with side chains including chemically active double bonds [3]. *DC* value of light-cured materials is affected by many factors, among which the most important are: the chemical structure and quantitative composition of monomers, the effectiveness of photoinitiators, the type and content of the filler, the translucency of the material, the distance, the intensity and the spectral distribution of the light source used for initiating polymerization as well as the environment in which the process occurs [3, 5, 8–10, 17].

The aim of the study was to assess *DC* value of four light-cured orthodontic adhesive systems.

EXPERIMENTAL PART

Materials

The study assessed four commonly used orthodontic adhesive systems from Polish distributors destined for sale in the European Union. The materials evaluated in the study and the composition declared by the manufacturers are shown in Table 2.

Methods of testing

The Fourier transform infrared spectroscopy (FT-IR) method was used to assess the number of double bonds in samples of four orthodontic adhesive materials. The authors used the Nicolet IS 10 spectrometer (Thermo Scientific, USA) with a Smart Orbit attachment equipped with a diamond crystal.

Five samples of each tested adhesive system were prepared, which were then placed in a Teflon matrix 5 mm in diameter of and 2 mm deep. Then unpolymerized material was removed from the matrix and placed on the spectrometer's diamond crystal to form a layer 1 mm thick. The samples were exposed to infrared radiation, wavenumber range 4000–400 cm⁻¹, 32 scans, then the spectrum of the reflected radiation was recorded. After the measurement, the material was placed between two sheets of polypropylene film and with a Teflon plate formed into a one-millimeter-thick layer, which was polymerized with a beam from LED 55 Curing Light (TPC Advanced Technology Company, USA) for 20 seconds from a distance of 5 mm. Such polymerized disc of adhesive material was freed from between the layers of foil and left at room temperature for one hour. The cured samples of adhesive systems were then placed on the spectrometer's diamond crystal and for each of them the spectrum of reflected infrared radiation was recorded again.

International abbreviation	Full chemical name			
UDMA	1,6-bis(methacryloxy-2-ethoxycarbonylamino)-2,4,4-trimethylhexane			
Bis-GMA	2,2-bis[4-(2-hydroxy-3-methacryloxypropoxy)phenylene]propane			
Bis-EMA	2,2-bis[4-(2-hydroxy-3-methacryloxyethoxy)phenyl]propane			
HEMA	2-hydroxyethyl methacrylate			
DEGDMA	diethylene glycol dimethacrylate			
TEGDMA	triethylene glycol dimethacrylate			
PEGDMA	poly(ethylene glycol) dimethacrylate			

T a ble 1. Chemical names and abbreviations of substances most commonly used for production of dental adhesive materials

T a ble 2. Orthodontic adhesive systems evaluated in the study and the content of individual methacrylate resins, fillers and catalysts of polymerization reaction declared by the producer

Trade name	Basic ingredients Filler content		Producer	
Contec LC	17–19 wt % of Bis-GMA 22–23 wt % of TEGDMA	silicates	Dentaurum GmbH & Co. KG, Germany LOT: 90370	
Resilience Light- -Activated Orthodontic Adhesive System	Bis-GMA TEGDMA camphorquinone	no data	Ortho Technology, Inc. Tampa, Florida, USA LOT: H002658	
Transbond Plus Color Change Adhesive	5–15 wt % of PEGDMA 5–15 wt % of 1,2,3-propanetricarboxylic acid 2-hydroxy- reaction products with 2-isocyanatoethyl methacrylate 2 wt % of Bis-GMA	35–45 wt % of silane treated glass 35–45 wt % of silane treated quartz < 2 wt % of silane treated silica	3M Unitec, Monrovia, Kalifornia, USA LOT: N686102	
Transbond XT Light Cure Adhesive Paste	45–55 wt % of Bis-GMA 45–55 wt % of TEGDMA <1 wt % of triphenylantimony <0.5 wt % of 4-(dimethylamino)-benzeneethanol <0.3 wt % of D,L-camphorquinone <0.03 wt % of hydroquinone	no data	3M Unitec, Monrovia, Kalifornia, USA LOT: N619082	

All tested samples were stored in separate containers in the dark at room temperature for 24 h, then once more the spectrum of the reflected radiation was measured.

The values obtained from the measurements described above were used to estimate the degree of conversion for each of the five samples of the four tested orthodontic adhesive systems in two time intervals, *i.e.*, 1 and 24 h after initiation of the polymerization process by visible light. The degree of conversion (*DC*) was calculated using the formula [6, 10, 14, 17]:

$$DC = \left(1 - \frac{R_t}{R_0}\right) \cdot 100 \% \tag{1}$$

where: $R_{0'}$, R_t – the ratios of peak areas [18, 19] for the wavenumbers 1638 and 1720 cm⁻¹ determined before and after polymerization during time *t*, respectively.

Wavenumber 1638 cm⁻¹ corresponds to the absorbance value of the aliphatic C=C double bonds, whereas wavenumber 1720 cm⁻¹ to the absorbance value of the C=O carbonyl bonds, constituting a reference value [3, 20, 21].

Statistical analysis

Statistical analysis of the obtained results was performed using Statistica 8.0 software package. In the process of hypothesis testing, the level of significance was assumed at α = 0.05.

The analysis regarded *DC* values of four studied materials at three points in time: prior to the initiation of polymerization, 1 h after exposure, and 24 h after exposure. For each variable arithmetic mean value, standard deviation, minimum value, median, maximum value were calculated. For the analysis of means, the analysis of variance model was used with repeated measures. The Newman--Keuls test was used as multiple test. On the basis of its results, homogeneous groups were distinguished.

RESULTS AND DISCUSSION

The results of *DC* investigation of the tested adhesive systems with respect to observation time are listed in Table 3.

Material	<i>t,</i> h	Number of samples	Mean value of DC	Standard deviation	Minimal value of DC	Median value of DC	Maximal value of DC
Contec LC	1	5	39.81	3.85	37.12	38.76	46.49
Resilience	1	5	53.07	8.77	37.49	56.10	58.59
Transbond Plus	1	5	30.12	6.74	22.11	33.92	36.53
Transbond XT	1	5	41.21	2.71	37.44	41.65	44.48
Contec LC	24	5	60.17	5.59	50.88	62.09	65.69
Resilience	24	5	68.10	4.32	60.58	69.57	71.13
Transbond Plus	24	5	49.19	3.42	43.85	49.10	52.24
Transbond XT	24	5	50.39	2.27	46.99	50.80	53.26

T a ble 3. Statistical analysis of results of conversion degree (DC) for the orthodontic adhesive systems evaluated in the study according to the period of observation

The values of *DC* of the studied orthodontic adhesive systems observed 1 h after initiation of the polymerization process equaled 30.12 % for Transbond Plus resin and 53.07 % in the case of Resilience material. Statistical analysis performed at the significance level of α = 0.05 showed that Contec LC and Transbond XT orthodontic adhesive systems demonstrated a comparable *DC* values (*p* > 0.05), which, however, was significantly higher (*p* > 0.05) than *DC* of Transbond Plus and significantly lower (*p* < 0.05) than value for Resilience.

DC after 24 h of curing of the evaluated samples ranged from 49.19 % for Transbond Plus to 68.10 % for Resilience. After 24 h of storage *DC* of Transbond XT samples was not statistically different (p > 0.05) from the value for Transbond Plus system.

Statistical analysis showed that the degree of crosslinking increased significantly (p > 0.05) for each material with the passage of time of observation. In addition *DC* values of Transbond XT and Transbond Plus materials obtained after 24 h were comparable to that recorded after 1 h of observation of Resilience adhesive samples.

Assessment of the effectiveness of the polymerization process in case of composite resins used in dentistry, performed by measuring DC of double bonds with FT-IR spectroscopy, is a method used in numerous studies [1, 7, 9–11, 13, 14, 17, 19, 20, 22, 23]. Available literature reports that DC values of orthodontic adhesive systems polymerized with visible light ranged from 22 to 88.24 % [1, 7, 9-11, 13, 14, 19, 20, 22]. Differences in DC of polymer matrix appear to result primarily from different chemical structure of individual composite materials used in dentistry. This thesis is confirmed by many investigators [1, 8, 9, 14, 18]. The quoted authors describe the presence of statistically significant differences in *DC* of various orthodontic adhesives while maintaining a uniform research methodology and photopolymerization conditions. Similar variations in the degree of crosslinking of individual materials polymerized in the same conditions were described by authors evaluating composite materials used in conservative dentistry [17, 23]. The quoted authors explained that the differences in the degree of crosslinking of the polymer matrix resulted from different chemical composition of each material, i.e., from the use of various primary and secondary monomers, inorganic fillers, as well as different concentrations and types of catalysts used in the polymerization reaction [3–6, 8, 17]. This thesis is confirmed by the results of the present study, during which all evaluated materials were subjected to identical polymerization protocol while orthodontic adhesives differed significantly (p > 0.05) in *DC*, both in relation to the type of material and the observation period.

In a study by Çörekçi *et al.* [9], the quoted authors evaluated *DC* of five orthodontic adhesives polymerized with visible light at 1200 mW/cm² intensity through a glass plate 3 mm thick. After measurements taken immediately after polymerization of orthodontic adhesives the researchers observed that for Transbond XT *DC* value was 83.09 %, and after 30 days of sample incubation in artificial saliva it decreased to 77.6 %. The authors, who used the FT-IR spectroscopy method for the assessment of crosslinking in Transbond XT orthodontic adhesive system, reported significantly higher conversion level than that observed for the same material in the present study.

Amato et al. [11] assessed DC values of orthodontic adhesives irradiated from a distance of 1 mm through polyester tape and glass barriers with a thickness of 1 mm. The authors evaluated *DC* for a combination of different times and irradiation power levels with a similar level of total energy supplied to the sample material by polymerization light, i.e., from 4.5 to 4.56 J. For Transbond XT orthodontic adhesive the reported average DC value was 64 %, whereas for Transbond Plus it equaled 87.2 %, in addition *DC* for each of the studied materials decreased with shortening of the exposure time and with an increase of the curing light's power. The values of *DC* reported by the quoted authors were higher than the values observed in the present study for the same orthodontic adhesive systems. In the case of Transbond XT after 1 h of observation it amounted to 41.2 % and after 24 h to 50.38 %, and for Transbond Plus material the values were 30.12 and 49.19 %, respectively. It seems that a lower conversion level of Transbond XT and Transbond Plus resins observed in the present study could be ascribed to a longer distance between the curing light's fiber and the surface of the material, which in the present study was 5 mm, as compared to 3 mm in the study by Çörekçi *et al.* [9] and to 1 mm in the study by Amato *et al.* [11]. A longer distance from the optical fiber could cause that less energy was transmitted to the mass of the material, resulting in lower efficiency of the polymerization process in the evaluated orthodontic adhesive systems.

Cerveira et al. [22] in their study applied the same method of adhesive sample preparation as in the present study, and they performed polymerization with the use of a halogen lamp with light intensity of 638 mW/cm² for 10, 20 and 30 s, and with a LED light at 450 mW/cm² for 5, 10 and 15 s. The authors noted that DC of Transbond XT was similar irrespective of the type of lamp used and ranged from 38.97 to 47.24 % for the LED lamp, and from 43.42 to 46.12 % for the halogen lamp. Purushothaman et al. [1] evaluated, among other things, DC of Transbond XT after 20 s of curing by LED lamp with light intensity of 1100-1200 mW/cm². The test conditions included irradiation of adhesive applied to a metal orthodontic bracket at the distances from the tip of the lamp of 0, 5 and 10 mm. For the distance of 0 mm the average value of *DC* equaled 26.46 %. After 40 s of polymerization with a halogen light at 450–510 mW/cm² from a distance of 0 mm, the same authors observed an average DC value for Transbond XT at the level of 25.50 %. In a similar study conducted by Sunitha et al. [7] the authors reported DC of Transbond XT material of 27.47 %. Both teams confirmed that increasing the distance between the tip of the lamp and the polymerized material resulted in a significant decrease in DC value of the assessed orthodontic adhesives.

Eliades *et al.* [19] published the results of their study in which they evaluated *DC* of Transbond adhesive, polymerized on the surface of ceramic and metal orthodontic brackets. The light was directed perpendicular to the brackets or onto their incisal and gingival edges. The authors reported that for several types of aesthetic brackets *DC* was similar regardless of the curing method, while in the case of metal brackets the observed values were 22 % for irradiation through the bracket and 48 % when the light fell on the edges of the brackets.

Shinya *et al.* [24] assessed *DC* of Transbond XT orthodontic adhesive after polymerization with LED light at 920 mW/cm² placed under a stainless steel bracket or irradiated directly. The results of 32.4 and 54.7 %, respectively, confirm that a metal bracket constitutes a barrier to the lamp light, which results in a significant decrease in *DC* of the orthodontic adhesive placed underneath.

The results of the reported studies indicate that in clinical conditions, where it is necessary to irradiate adhesive material located under the base of the bracket, *DC* of the polymerized adhesive system can be significantly lower than that observed in laboratory studies, in which the bonding system is polymerized through direct exposure to visible light from the lamp.

This observation is not confirmed in the study by Jagdish *et al.* [18] evaluating the polymerization process of Transbond XT material in conditions simulating clinical environment. The quoted authors reported an average *DC* value of the adhesive located under the bracket's base at 48.7 %, which was similar to that observed in the present study.

A large range of conversion levels reported by various authors for the same types of light-cured materials can be explained by using different methodology for evaluation of the polymerization process and by different conditions in which the crosslinking processes were initiated. *DC* value of dental materials based on polymers is affected not only by the kind and intensity of the light source, but also by the exposure time, the distance of the material from the lamp, the presence or absence of additional barriers between the light source and the material, the glancing angle of the light beam, and the environment in which the polymerization is performed.

CONCLUSIONS

The results of the present study confirm the thesis according to which the polymerization process of dental composite materials is not complete. Chemical instability of partially polymerized orthodontic adhesives can not only have a negative impact on their clinical characteristics, but also pose a potential threat to the health of patients. Components of composite materials used in dentistry released into the oral cavity demonstrate, among other things, cytotoxic as well as mutagenic properties, and can cause abnormal development processes in fetuses [1, 4, 7, 9, 14, 18, 25–27]. In the light of this information it is necessary to set new standards of safety evaluation for medical materials and to develop orthodontic adhesive systems characterized by higher chemical stability than currently available products.

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