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Relevance of In Vitro Tests of Adhesive and Composite Dental Materials

A Review in 3 Parts

Part 1: Approval Requirements and Standardized Testing of Composite Materials According to ISO Specifications

Keywords: composite, flexural strength, depth of cure, color stability, radiopacity

Summary The first part of this three-part review on the relevance of laboratory testing of composites and adhesives deals with approval requirements for composite materials. We compare the *in vivo* and *in vitro* literature data and discuss the relevance of in vitro analyses. The standardized ISO protocols are presented, with a focus on the evaluation of physical parameters. These tests all have a standardized protocol that describes the entire test set-up. The tests analyse flexural strength, depth of cure, susceptibility to ambient light, color stability, water sorption and solubility, and radiopacity. Some tests have a clinical correlation. A high flexural strength, for instance, decreases the risk of fractures of the marginal ridge in posterior restorations and incisal edge build-ups of restored anterior teeth. Other tests do not have a clinical correlation or the threshold values are too low, which results in an approval of materials that show inferior clinical properties (e.g., radiopacity). It is advantageous to know the test set-ups and the ideal threshold values to correctly interpret the material data. Overall, however, laboratory assessment alone cannot ensure the clinical success of a product.

Introduction

Composite restorations are exposed to various influences in the oral cavity and must therefore meet high demands in terms of mechanical, chemical, and color stability. There are numerous methods for characterizing dental materials and testing them for clinical suitability. Many of these are used in dental product advertising to emphasize certain material properties, for instance, shrinkage values, bond strength, wear or abrasion, color stability, and others. The advertised material is usually compared with the competitor's products and the results are presented as bar diagrams including (rarely) scattering of the data. To the dentist, this suggests that the lower the shrinkage, or the higher the bond strength, or the less the dye penetration, or the lower the abrasion, the better the material is in clinical use.

The majority of the dental literature consists of results from laboratory tests, as this is much faster and less labor-intensive than collecting clinical data. The proportion of published clinical prospective studies compared to all other published studies in four international English-language dental journals from 2003 to 2008 ranged between 2% and 23% (Fig. 1).

Because conducting clinical studies is complicated and expensive, and results are available only years later, the easiest thing to do is refer to and use results from laboratory tests or simulations. But how much relevant information do such in vitro tests of dental materials really provide in terms of clinical suitability?

Laboratory test results have consistently provided the basis for recommendations on how dentists should use composite materials in their daily clinical routine. Then, years later, clinical studies discover that the restorations placed according

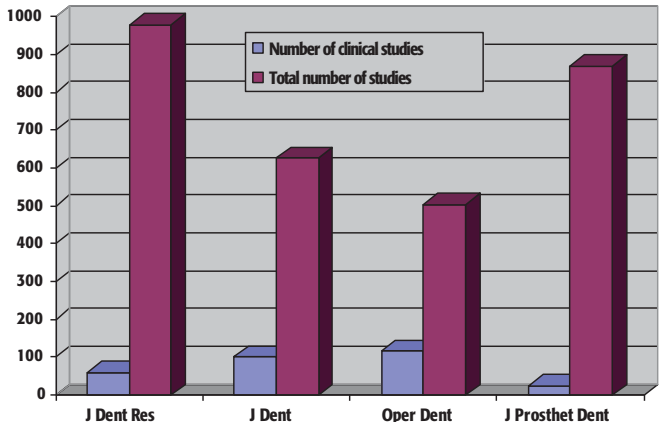

Fig. 1 Number of randomized clinical trials compared to total number of publications in four English-language dental journals (search period 2003–2008, search month 12/2008).

to these recommendations are no better than those performed with other, usually simpler techniques. Good examples of this are the use of translucent wedges and transparent matrices versus wooden wedges and metal matrices (Demarco et al. 2007, 2010), "selective" bonding versus "total" bonding (Baratieri & Ritter 2001, Brunthaler et al. 2003), or "with softstart polymerization" versus "without softstart polymerization" (Chan et al. 2008). One clinical study even questioned the incremental technique in posterior composite restorations, since the clinical results of fillings bulk-placed (one increment) in mid-sized cavities were comparable to the results of studies in which the incremental technique was used (Sarrett et al. 2006).

Materials and Methods

To answer the question about the relevance of laboratory tests, first the currently valid guidelines and test methods for composite materials were reviewed: the approval requirements of the medical device directive and CE certification, and the ISO test regulations for dental materials (Schorn 1994, ISO 1997, 1998, 2010). Subsequently, an electronic literature search was conducted in the databank PubMed. The articles evaluated were selected using the following keywords: "composite restoration", "survival rate and composite", and "composite restoration" combined with "flexural strength", "depth of cure", "color stability", "water sorption", "solubility", "radiopacity" and "biocompatibility".

Requirements for a Composite Material

A restorative material must restore the function (pulpal protection, chewing) and esthetics (anatomic shape, color) of dental hard tissue lost due to caries or trauma. With composites, malposition/malocclusion or gaps can also be corrected. Composites are additionally used to repair restorations, build up fractured teeth, and create stump build-ups for prosthetic reconstructions. Further, composite materials are implemented to cement indirect restorations and retaining elements of orthodontic appliances, and to seal fissures. The different application areas of composite materials place different demands on the material itself.

Ideally, the dentist should be able to simply and ergonomically handle the restorative material, which should also retain its function and esthetics as long as possible.

Today, most composites are sold as universal composites, i. e., the material is suitable for every indication and size of direct restoration. Hence, the composites must fulfill a broad spectrum of handling and load-bearing requirements due to the demands placed on it by the oral environment and mastication (Mjör 2007):

1. *Requirements for handling properties of the material.* High viscosity, packability, not sticking to instruments, flowability and polishability.
2. *Requirements for the esthetics of the material.* The composite restoration should not be visible at a social, speaking distance.
3. *Requirements for the longevity of the placed filling.* Flexural strength, fracture strength, surface hardness, water sorption, solubility, polymerization shrinkage and shrinkage force. Of course, the skill and experience of the dentist in addition to patient-related factors, such as caries activity and parafunctions, also influence the longevity of composite restorations.

Medical Device Directive and CE Certification

Most of the materials used in dentistry are subject to the *Medical Device Directive 93/42* (Schorn 1994), valid Europe-wide. The products are divided into four different classes. With the exception of the products in the highest class (Class III, e. g., bone cement), the law does not require clinical testing. The products must chemically and physically correspond to the harmonized international standards (ISO). Thus, for dental products (Class IIa, e. g., adhesive systems, composites, cements, fixed dental prosthesis materials; Class IIb implants), it is up to the manufacturer to decide with what sort of clinical documentation, if any, the product is placed on the market. Although clinical documentation for such products is necessary, it can be based merely on in vitro data and comparative literature with data from clinical studies on similar materials. The *CE mark* (CE stands for *Communautées Européennes*) is not a "seal of quality" for a specific product; it only means that the product corresponds to the basic requirements of the guidelines and harmonized standards. As of June 1998, all products brought onto the market in the EU or the European Economic Area and Switzerland must bear the CE mark.

ADA Guidelines

The *American Dental Association* (ADA) established guidelines for the awarding of a Seal of Acceptance. The ADA Guidelines (ADA 1993, 2001a, b) were discontinued for composites and adhesives at the end of 2008, because almost no dental companies still participated in the program (Berthold 2004). The ADA Guidelines demanded, in addition to the laboratory test, two clinical tests of the product over 18 months before awarding the product the Seal of Acceptance. This was the main reason that dental companies rejected the program, as clinical studies considerably delay the marketing of a product.

Requirements for a Laboratory Test

Laboratory tests are useful for testing new operative techniques and materials before they are clinically implemented. The methods employed, however, should meet the following requirements (FDA 1978):

1. The results must be reproducible, i. e., when the same test is repeated under the same conditions and with the same materials, the same results should be obtained.
2. The parameters which influence the test results must be known.

3. The variability of the measured values must be low and within an acceptable range. The coefficient of variation, that is, the ratio of the standard deviation to the mean, should be under 20%. The coefficient of variation determines the number of specimens per group.
4. If devices are employed for the test itself and/or to measure parameters and post-testing conditions of the specimens, then these devices must be suitable for the given purpose, that is, they must be qualified. This, in turn, must be proven and documented. A device may have to be calibrated before performing the test or measurement.

If all of these requirements are met, the test method is *internally valid*. Because the test method is used to provide information or prognoses about the clinical suitability of the material, the results must correlate with clinical findings. If this is the case, the test is also *externally valid*.

These requirements were described for medical devices and compiled under the name "Good Laboratory Practice" by quality-control authorities such as the Food & Drug Administration (FDA) in Washington or the European authorities in Brussels in the 1970s and 1990s, respectively (EU 1993, FDA 1978). These specifications apply to medical devices in general and are not defined specifically for dental materials.

ISO Standard

A plethora of standards and technical specifications exists for dental materials. The advantage of these standards is that defined test methods are described which can be reproducibly performed with relatively easily accessible means in laboratories. The specifications for material properties are the greatest common denominator between the representatives of industry, authorities, and universities, who work together in the standardization committees. As a consequence, the limits can sometimes lie in the suboptimal range, which will be shown below. The *International Organization for Standardization ISO* ("isos": Greek for "equal") has to date set over 17,500 international standards in a great variety of fields.

For dental composites, the valid standard is ISO 4049 "Dentistry – Polymer-based filling, restoration, and luting materials" (ISO 2009a). This standard describes both testing methods and "minimal requirements" (Table I). The spectrum of properties of approved dental products is relatively wide.

In the following, the most important laboratory tests according to ISO are presented and discussed in terms of their clinical relevance.

ISO 4049

Depth of Cure

The depth of cure determines how thick the composite layer can be and still attains adequate conversion. In the test, composite is placed in the hole (6 mm long × 4 mm) of a stainless steel mould and polymerized, the unpolymerized portion is removed with a plastic spatula, and the remaining composite thickness is measured and divided by two. Composites usually present a depth of cure of at least 2 mm. The ISO standard specifies a minimum of 1.5 mm (ISO 2009a).

Another method of determining depth of cure is to measure the Vickers hardness of the top and bottom of specimens of different thicknesses. In this method, a pyramid-shaped diamond instrument with a defined speed and a load of ca. 10 N is pushed into the material and the diameter of the diamond impression in the material recorded. A material qualifies as completely cured when the surface hardness of the bottom is at least 80% of that of the top. Studies have shown that this value correlates well with half the depth of cure test as specified in the ISO standard (Tsai et al. 2004).

Whether a composite polymerizes completely also depends on other factors, for instance, the transparency of the composite, the power density of the polymerization unit, the duration of irradiation, and the distance between the emission window of the light probe and the composite to be polymerized (Krämer et al. 2008). In Class II cavities with deep proximal boxes reaching into the dentin, the first increment of composite at the gingival floor must be irradiated with a device providing 560 mW/cm^2 for at least 40 s. If a unit emitting 1200 mW/cm^2 is used, 20 s of polymerization suffices to completely harden the material (Ernst et al. 2004). Besides a variable and unreliable bond between the adhesive system and the substrate, inadequate conversion of the composite can also be responsible for the frequent occurrence of secondary caries in the gingival part of Class II restorations. This site has been evaluated as a critical area for the formation of secondary caries: about 80% of all secondary caries forms at the cervico-gingival margin of Class II fillings and only 20% at the occlusal margin (Mjör 1998) (Fig. 2). Moreover, marginal discoloration is found much more frequently in this area than elsewhere (Wilson et al. 2006).

Tab. I Physical tests of dental composites according to ISO standard 4049 (ISO 2009a) and their clinical importance. The ISO standard threshold values are given along with threshold values formulated by the authors as characteristic of a good dental composite.

Physical test according to ISO 4049	Threshold value according to ISO 4049	Threshold value for clinical suitability	Clinical importance
Depth of cure (mm)	⩾ 1.5	>2	Stability and integrity
Sensitivity to ambient light (sec)	⩾ 60	120–240	Handling time
3-point bending test (MPa)	⩾ 80	⩾ 90–100	Mechanical stability
Water sorption (µg/mm^3)	⩽ 40		Chemical stability, Expansion
Solubility (µg/mm^3)	⩽ 7.5		Chemical stability Biocompatibility
Color stability	No change		Color stability
Radiopacity (%Al)	⩾ 100	⩾ 200	Distinction between restoration and tooth/caries

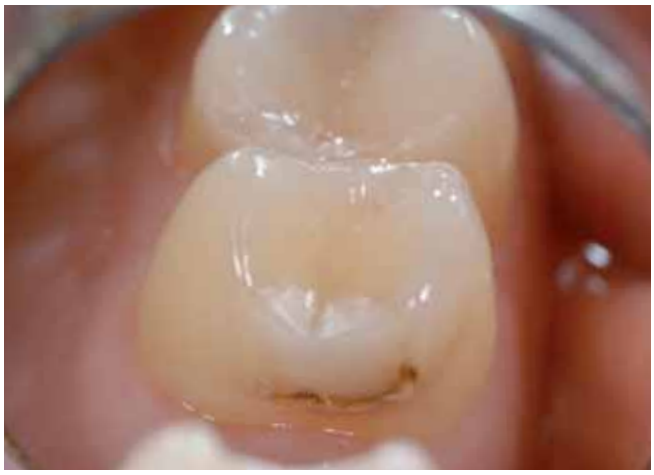

Fig. 2 Secondary caries at the distal gingival floor of a Class II composite restoration in a premolar.

In addition to insufficient polymerization duration, another possible source of error is inadequate maintenance and monitoring of the power density of light-curing units in private practices. In a field test of 301 dental offices in Germany, 26% of the units emitted less than the minimum required power density of 400 mW/cm² (Ernst et al. 2006, Rueggeberg et al. 1994). Furthermore, complaints had to be made for 48% of the light probes as they exhibited defects or composite residuals were stuck to them (Ernst et al. 2006).

The direct clinical relevance of the depth of cure test remains questionable, not least because access for polymerization in the oral cavity is difficult and the light probe usually does not come into direct contact with the restoration surface. Additionally, polymerization is often performed by an assistant, which can lead to further imprecision in curing.

Sensitivity to Ambient Light

The clinical relevance of this test lies in the information about how long the dentist can handle the material before the ambient light cures it. The ISO standard stipulates a handling time of at least one minute. In the test, appr. 30 mg of test material is illuminated for 60 s under predetermined conditions (8000 lux ± 1000) with a UV filter. The material is then compressed between two glass plates to a thin film which may not exhibit any inhomogeneities due to premature polymerization. In the clinical situation, premature polymerization of restorative composites (as opposed to luting composites) is usually not a problem if the operator is experienced. If a product is advertised as having a long handling time, it is important to bear in mind that such products also tend to have a longer polymerization time, since the curing reaction is considerably delayed through the given composition of initiators and inhibitors (Ilie & Hickel 2006).

Flexural Strength

The flexural or bending strength is a measure of the fracture resistance of a material. For restorative materials in occlusion-bearing areas, the ISO standard demands a flexural strength of at least 80 MPa. For this test, bar-shaped specimens (25×2×2 mm) are made, stored in water for 24 h and at 37 °C, and loaded until failure in a univeral testing machine (crosshead speed 0.75 mm/min [+/–0.25]). The flexural strength (BF) in the 3-point bending test is calculated with the following formula: $BF = 3F\ d/2wh^2$ (F = maximum force, d = distance between the two anchors, w = width of the specimen, h = height of the specimen).

Because the flexural strength changes after water storage, the value at 24 h only provides limited information. Reliable data on the behavior of the material are obtained when the value after 1 day's storage is compared to that after 1 month of water storage.

Composite materials with a flexural strength less than 80 MPa – the minimum given in the standard – showed increased fractures in clinical studies. In clinical tests of the composite material Solitaire (Heraeus Kulzer), which was put on the market in 1998 (flexural strength: 57 MPa) (Adabo et al. 2003), over 20% of the Class II restorations exhibited fractures in the area of the marginal ridge and margins after only 2 years (Fig. 3) (Ernst et al. 2001; Krämer et al. 2005). The manufacturer altered the material, which then possessed a flexural strength of 120 MPa. The subsequent clinical studies showed that Class II restorations with Solitaire 2 exhibited much fewer restoration fractures after 2 years (Burke et al. 2005, Gallo et al. 2005). A similar relation between flexural strength and fracture resistance of composite materials was found for the indication of incisal build-up (Class IV). Where 42% of incisal build-ups made of Durafill (Heraeus Kulzer) fractured within 3 years, only 5% of the Estilux (Heraeus Kulzer) restorations did so (Tyas 1990). Here, too, the reason was low flexural strength: Durafill showed a flexural strength of 70 MPa and Estilux 120 MPa.

For restorations exposed to greater mechanical loads, e.g., Class II and IV fillings, the mechanical stability is highly important. Ideally, the minimum flexural strength is 90–100 MPa, especially because some composites exhibit diminished flexural strength after longer water storage or thermocycling (Janda et al. 2006). The flexural strength thus possesses a direct clinical correlation and great predictive value for the success of a material in practice.

Water Sorption and Solubility

The standardized test for water sorption stipulates that a standardized specimen be stored in a desiccator at 37 °C for 22 h, followed by 2 h more at 23 °C. The material is then weighed. This is repeated until the weight changes by no more than 0.1 mg. This weight is then recorded as the initial weight m_1. Subsequently, the dimensions of the specimen are measured to determine the volume (V) after the drying phase. Then the specimen is stored in water at 37 ^{0}C for 7 days. The specimen is weighed again after water storage (m_2) and dried again as at the beginning of the test. When the weight of the specimen

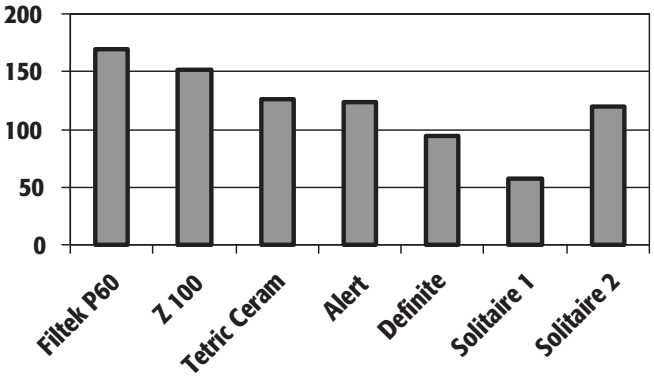

Fig. 3 Flexural strength (MPa) of 7 composite materials tested according to ISO standard 4049 (after 24-h water storage) (Adabo et al. 2003).

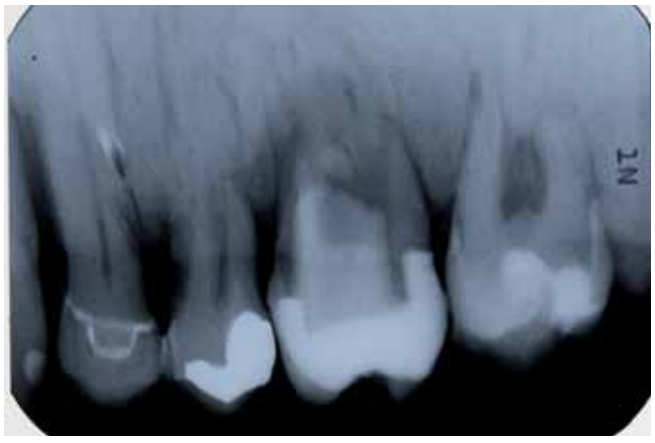

Fig. 4a The restorations exhibit different radiopacities. Note that the mesial restoration at tooth 27 is barely distinguishable from the dental hard tissue.

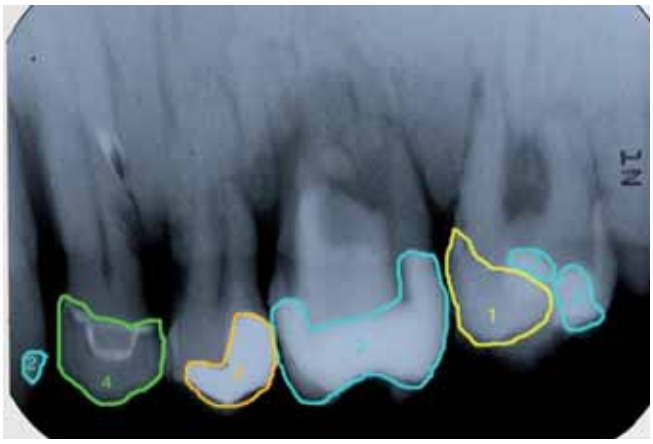

Fig. 4b 1) Siloran (Filtek Siloran), 2) nanohybrid composite (Ceram-X), 3) amalgam (Oralloy), 4) glass ceramic overlay (Empress CAD) cemented with a composite resin (Variolink II).

remains constant, the weight m_3 is determined. From these values, the water sorption W_{sp} can be calculated with the following formula:

$W_{sp} = (m_2 – m_3) / V$ (m^2: weight after water sorption, m^3: weight after re-drying the specimen). For the ISO standard to be met, a value of less than 40 µg/mm³ must be reached.

For the solubility test, the previously recorded values can be used in the following formula to calculate the solubility W_{sl}:

$W_{sl} = (m_1 – m_3) / V$ (m_1: weight of specimen after initial drying, m_3: weight of specimen after water sorption and re-drying). This value may not exceed 7.5 µg/mm³.

High water sorption negatively influences the swelling, discoloration, and transparency of the material (Dietschi et al. 1994). During water sorption, first the bond between the matrix and the filler is weakened, and finally the material strength is decreased by the accumulated water per se (Toledano et al. 2003). The consequence of this material alteration is that other measured parameters, e.g., tensile and flexural strength, abrasion resistance, also undergo detrimental changes (Ferracane et al. 1998, Toledano et al. 2006, Sideriou et al. 2003, Swartz et al. 1982, Momoi & McCabe 1994, Sarrett & Ray 1994, Asaoka & Hirano 2003). Water sorption tests according to the ISO standard yielded 27 µg/mm³ for Admira and 12 µg/mm³ for Tetric Ceram (Janda et al. 2007), both lower than the ISO limit and therefore suitable for clinical use. Admira and Tetric Ceram did not differ significantly in the 5-year clinical comparison (Bottenberg et al. 2009). The clinical relevance of this test appears established.

Shade and Color Stability

To determine color stability in vitro according to the ISO standard, a first specimen is stored dry for 7 days at 37 °C, and serves as the color reference. A second specimen is stored in water for 7 days also at 37 °C, to demonstrate which color changes arise as a result of water storage. A third specimen is initially dried at 37 °C for 24 h. Then, one half of the specimen is covered with foil, and the whole specimen is stored in water (37 °C) in a light box (xenon light). After 24 h, the foil is removed and the specimen is dried for another 5 days (37 °C). Specimen number 2 is compared with the manufacturer's color ring. No notable deviations in shade compared to the color ring should be visible. In addition, the specimen must be evenly pigmented, so that optically, without magnification, no color differences are perceptible. The color congruency of the second and third specimens is compared with that of the first. No electronic devices are used to measure the color, which may seem confusing at first, as all other parameters are standardized. Nevertheless, the human eye is still considered to yield the most highly reproducible color assessment; electronic devices exhibit a certain degree of imprecision (Hugo et al. 2005).

Radiopacity

Radiopacity is set by comparison to an aluminum standard. A standardized specimen of the composite material is x-rayed together with the aluminum standard (65 ± 5 kV, exposure duration 0.3–0.4 s). The distance between the x-ray tube and the film is 300–400 mm. The optical density of the test material is then compared to the aluminum standard, and must be greater than or equal to that of the standard.

Radiopacity is an example of the suboptimal limits set by the ISO standards. The minimum value of 100%Al is too low for clinical use. A composite material must have a radiopacity of at least 200%Al to be distinguishable from dental hard tissues (Espelid et al. 1991) (Figs 4a, b). This shows that although the test protocol is clinically relevant, the minimum value is set too low.

Result Discrepancies between Testing Laboratories

Values differing by 10–15% of the mean lie within the range of variation of the test and are determined by the material, the test methods, and the manual fabrication of the specimens. Deviations greater than this, however, can have different causes:

- The operator knows the reference material and its application better than the new material to be tested.
- The tested product differs slightly in terms of composition from the definitive formulation of the material on the market.
- Production batches differ slightly among themselves, but this must remain within certain limits.
- The values measured by certain test methods, e.g., flexural strength, depend greatly on the quality and surface treatment of the specimens (Huysmans et al. 1996).

In addition to the test standards mentioned above, there are also ISO tests for the biocompatibility of restorative materials

(ISO 2008, 2009b). These standards are the subject of substantive discussions. Thus, these methods are not further described here.

Conclusions

At first, the laboratory tests according to ISO seem to be very far from clinical reality. None of the test set-ups use natural teeth, but instead manufacture standardized material specimens. A great advantage of standardized testing is that the values obtained in different institutes can be compared to each other. Furthermore, these in vitro tests provide physical values that are of crucial importance for assessing the clinical suitability of the materials. This is particularly true for flexural strength. The disadvantage of the standards, however, is that some of them lie below the ideal range, because the limits are based on a consensus between the manufacturers and the testing institutes.

The standardized laboratory tests are important for the first material analysis. As opposed to other test methods, they correlate well in part with the clinical data (Table II). Laboratory tests do not replace clinical tests, but they do increase the safety of the patients who participate in controlled studies of the new materials. However, if the manufacturers rely solely on the ISO standard tests before putting their products on the market, this may lead to unexpected problems in clinical use, problems which could not be anticipated in the laboratory tests. This is especially valid for innovative material concepts for which no analogous comparisons to existent systems can be drawn. For this reason, clinical studies of appropriate duration are important.

Tab. II Overview of the common in-vitro methods for testing dental materials and adhesive systems and their clinical relevance

Test	Standardized test protocol	Test validated	Clinical relevance
Flexural strength	yes	yes	yes
Depth of cure	yes	yes	yes
Water sorption	yes	yes	yes
Solubility	yes	yes	yes
Color stability	yes	yes	yes
Sensitivity to ambient light	yes	yes	moderate
Radiopacity	yes	yes	yes

Résumé

La première partie de cet aperçu sur la pertinence des tests de laboratoire sur les composites et systèmes adhésifs relate des dispositions d'autorisations pour matériaux composites. La pertinence des résultats de laboratoire est discutée à partir d'une littérature choisie. Les tests standards normés ISO sont présentés et discutés tout en mettant principalement l'accent sur les tests physiques. Ces tests comportent des mesures de la résistance en flexion, de la profondeur de polymérisation, du temps de travail, de la sensibilité à la lumière environnante, de la stabilité de la couleur, de l'absorption d'eau et de dissolution, ainsi que de la radio-opacité. Certains tests tels que celui de la résistance en flexion ont montré une corrélation directe avec la clinique. Ainsi, des fractures marginales sur des restaurations postérieures ou des fractures d'angles incisifs ont été plus souvent observées lorsque la valeur minimale ISO requise de résistance en flexion n'était pas atteinte. Pour d'autres tests, la corrélation clinique fait défaut ou encore le seuil minimal requis du test ISO est trop bas, ce qui permet à certains matériaux d'être autorisés tout en démontrant des insuffisances cliniques (par exemple pour la radio-opacité). Il est dès lors avantageux de connaître les dispositions des tests ainsi que les valeurs idéales des propriétés des matériaux composites afin d'interpréter correctement les informations sur un produit donné. Au final, il est à relever que les tests au laboratoire ne suffisent pas pour garantir le succès clinique.

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