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Effect of Cartridge Storage Time and Ambient Laboratory Conditions on the Stability of Mechanical Properties of Bis-Acryl Interim Resin Materials

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Shelf life; storage time; properties degradation; premature aging; bis-acryl interim resin materials.

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Abstract

Purpose: To investigate the stability of mechanical properties during the entire shelf life of chemically-activated bis-acryl interim resin materials stored at ambient laboratory conditions.
Materials and methods: Four bis-acryl interim resin materials with no explicit recommendation for refrigerator storage were selected (Luxatemp Plus, Visalys Temp, Structur 3, Structur Premium). Following cartridge storage at ambient laboratory conditions in 6-month intervals, 30 specimens ($2.0 \times 2.0 \times 25.0$ mm^3) were prepared in each case and stored in distilled water (37.0 ± 1.0 °C) with 3 different water storage times (2 hours, 24 hours, 7 days). Flexural strength, modulus of elasticity, modulus of toughness, and elastic recovery were determined (3-point bend test). Data were subjected to a three-way multivariate analysis of variance (MANOVA) to determine overall significance, followed by a series of separate one-way ANOVAs after data were split, using Tukey and Games-Howell post hoc tests to identify significant differences between groups ($\alpha = 0.05$).
Results: While Luxatemp Plus showed no significant decrease for all mechanical properties during the investigated shelf life of 21 months ($p > 0.05$), Visalys Temp exhibited a continuous decline of the corresponding values ($p < 0.05$), irrespective of the water storage time. Structur 3 and Structur Premium properties remained almost unchanged during the first 6 months of the examination period. Subsequently, a progressive decrease was observed for flexural strength and modulus of elasticity, while most modulus of toughness and elastic recovery results revealed little alteration.
Conclusion: Depending on the bis-acryl interim resin material, mechanical properties appeared to be susceptible to continuous degradation processes during cartridge storage at ambient conditions, especially towards the end of the material shelf life. For some materials, the chemical stabilization and recommended storage conditions should be revised by the manufacturers to prevent premature aging during shelf life.

Unlike definitive restorative materials, interim resin materials (IRM) are only intended for use for a comparatively short time in the oral environment to protect prepared teeth from chemical and bacterial irritations.[1] Consequently, little scientific data on the clinical performance of IRM is currently available.[2–7] However, their mechanical properties determined in vitro have been adequately discussed and compared.[8–13] Although in such material comparisons, computer-aided design and computer-aided manufacturing (CAD/CAM) fabricated IRM reveal a superior fit and higher fracture resistance in vitro,[14–16] many dentists continue to use the well-established, direct techniques as a reliable alternative. Therefore, in many dental practices, large stocks of IRM cartridges can be found stored in drawers, and manufacturers permit the storage of their materials at room temperature without explicitly recommending the use of a refrigerator.[17,18] Apparently, initiation systems have been specifically optimized for IRM, which then results in less sensitivity to variable laboratory temperatures and possibly little impairment of shelf life properties.

However, an early shelf life survey to define the extent of material problems related to the storage stability of dental materials indicated that a considerable quantity of clinically applied dental materials was very close to or even older than the expiry date stated by the manufacturer.[19] Furthermore, deterioration of perishable materials was believed to be a significant problem in dental practices.[20] With this in mind, stability concerns related to the useful life of sensitive restorative materials still appear to be important today.[21–25] The common trend of

Table 1 Tested bis-acryl interim resin materials

Material	Code	Manufacturer	MR[a]	Color	Lot No.	Expiry Date	Maximum shelf life[b]	Recommended storage conditions[c]
Luxatemp AM Plus	LT	DMG, Hamburg, Germany	10:1	A3.5	708746	03/2016	2 years	15 °C to 25 °C, dry place, no refrigerator
Visalys Temp	VT	Kettenbach, Eschenburg, Germany	10:1	A2	130811-27	09/2015	2 years	18 °C to 25 °C, dry place, no refrigerator
Structur 3	ST3	Voco, Cuxhaven, Germany	1:1	A3	1403332	03/2016	2 years	4 °C to 23 °C
Structur Premium	STP	Voco, Cuxhaven, Germany	1:1	A3	1407405	03/2016	2 years	4 °C to 23 °C

[a] MR, Mixing ratio base : catalyst (by volume).

[b] Counted from the date of cartridge filling; information provided by the manufacturer.

[c] Information from user instruction.

allowing two-component composite resins to be stored at room temperature without refrigeration is well accepted and extensively promoted. However, some current studies show that these materials, which may still contain apparently vulnerable initiators and other sensitive chemicals, are prone to changes in properties induced by temperature[23,24] and storage time effects later in their shelf life.[25] This calls into question the performance of IRM during their shelf life if they are not refrigerated. The authors know of no studies that provide reliable information about the effect of cartridge storage time at ambient laboratory conditions on mechanical properties during the useful life of IRM. In general, the impact of temperature and storage time on premature aging until the last month of the shelf life has scarcely been evaluated. Therefore, the aim of the present study was to investigate the change in flexural strength (FS), modulus of elasticity (ME), modulus of toughness (MT), and elastic recovery (ER) of different IRM that were stored at well-defined ambient laboratory conditions during their shelf life. The null hypothesis to be tested was threefold: the mechanical properties are independent of (1) the cartridge storage time, (2) the tested IRM, and (3) the water storage time of cured specimens.

Materials and methods

Four commonly used IRM, which differed in chemical composition and base/catalyst mixing ratios, were tested. Materials were purchased commercially with 6 auto-mix cartridges of the same lot number and were carefully selected to ensure expiry dates that were as similar as possible (Table 1). Cartridges were used as received without further manipulation and immediately stored after receipt under carefully controlled ambient laboratory conditions (23 ± 1 °C; 50 ± 5% relative humidity)[26] in the same dark place. None of the manufacturers recommended storing the materials in a refrigerator (Table 1). Cartridge storage times were defined as 6-month intervals starting from the date where the cartridges of all IRM were available. The expiry month of the shelf life was assigned E and the first joint inspection date scheduled at 21 months before E was defined E-21m. All further inspection dates were assigned accordingly (E-15m, E-9m, E-3m) with the material tests performed at 15, 9, and 3 months before E (Fig S7).

A stainless-steel mold was used to prepare 30 bar-shaped specimens (2.0 ± 0.1 mm × 2.0 ± 0.1 mm × 25.0 ± 2.0 mm) at the scheduled inspection dates for each group in accordance with ISO 4049:2019.[27] Ten specimens from each of 3 cartridges with the same lot number were cured in line with the times recommended by the respective manufacturers. They were mixed, randomly selected and immediately placed in 3 separate prewarmed glass bottles already containing distilled water (37.0 ± 1.0°C, each with 10 specimens) as previously described[8,28,29] and stored in an incubator (Ehret, Emmendingen, Germany) at 37.0 ± 1.0°C for respectively 2 hours, 24 hours, and 7 days. The very short storage time of 2 hours was purposely chosen in the present investigation, because previous studies for bis-acryl interim resin materials during this time period revealed comparatively low mechanical properties[8,28] which have a high potential to facilitate the detection of likewise slight material changes. To avoid partial surface damage and impairment of the ongoing polymerization process, excess material on specimen surfaces was carefully removed at the earliest after incubation by grinding on wet silicon carbide abrasive paper (1000 grit, Leco Corp, MI) followed by simultaneous adjustment of the specimen dimensions as previously described.[8,28,29] Dimensions were controlled using a digit outside micrometer (Type 293-521-30, Mitutoyo, Kawasaki, Japan, accuracy ±1 µm) and the specimens were then loaded until rupture in a 3-point bend test with a newly calibrated universal testing machine (1454, Zwick/Roell, Ulm, Germany) and a 0.5-kN load cell.[30] The span width (L) between the two supports was 20.0 mm and the crosshead speed was 1 mm/min. Specimen deflection was detected via the crosshead motion of the testing machine, and engineering stress-strain curves were used for the modulus of toughness calculation without correction for true stress-strain material behavior.[31] FS (MPa),[26,27] ME (MPa),[26,28] MT

Table 2 Results of the three-way MANOVA with Pillai's trace

Independent variable	Value	F	Hypothesis *df*	Error *df*	*p*	Partial Eta squared	Observed power
Cartridge storage time	0.923	38.5	16	2052	<0.001	0.231	1.000
Water storage time	1.076	148.8	8	1022	<0.001	0.538	1.000
Material	1.480	124.6	12	1536	<0.001	0.493	1.000
Material × Cartridge storage time	1.216	20.4	44	2052	<0.001	0.304	1.000
Material × Water storage time	1.430	47.6	24	2052	<0.001	0.358	1.000
Cartridge storage time × Water storage time	0.378	6.7	32	2052	<0.001	0.094	1.000
Material × Cartridge storage time × Water storage time	0.646	4.5	88	2052	<0.001	0.161	1.000

p values <0.05 designate significant differences.

Table 3 Test of between-subjects effects for fixed factors cartridge storage time, water storage time, and material on mechanical properties (FS, ME, MT, ER)

Source	Dependent variable	Sum of squares	*df*	Mean of squares	*F*	*p*	Partial Eta squared	Observed power
Cartridge storage time	FS	22,812.9	4	5,703.2	652.0	<0.001	0.836	1.000
	ME	18,381,044.0	4	4,595,261.0	301.3	<0.001	0.701	1.000
	MT	99.4	4	24.9	26.4	<0.001	0.171	1.000
	ER	6.4	4	1.6	108.6	<0.001	0.459	1.000
Water storage time	FS	129,908.2	2	64,954.1	7425.6	<0.001	0.967	1.000
	ME	153,552,024.0	2	76,776,012.0	5033.2	<0.001	0.952	1.000
	MT	92.4	2	46.2	49.0	<0.001	0.160	1.000
	ER	22.7	2	11.4	772.6	<0.001	0.751	1.000
Material	FS	47,614.0	3	15,871.3	1814.4	<0.001	0.914	1.000
	ME	111,856,047.0	3	37,285,349.0	2444.3	<0.001	0.935	1.000
	MT	450.8	3	150.3	159.5	<0.001	0.483	1.000
	ER	3.0	3	0.995	67.7	<0.001	0.284	1.000

p values <0.05 designate significant differences.

(MJ/m^3),[32–34] and ER (MJ/m^3)[33–36] were calculated by using the following equations:

$$FS = \frac{3F_{\max}L}{2wh^2},\ ME = \frac{F_{lin}L^3}{4d_{lin}wh^3},\ MT = \frac{9A}{whL},\ ER = \frac{FS^2}{2ME}$$

where $F_{\max}$ (N) is the maximum load, L (mm) is the span width between the 2 support bars, and w (mm) and h (mm) are the width and height of the specimen. F_{lin} (N) is the force in the linear part of the stress-strain curve and d_{lin} (mm) is the corresponding deflection at F_{lin}. A (J) is the total area under the load-deformation curve (work performed by the applied load to deflect and fracture the specimen) obtained with the software used for the testing machine (TestXpert, release 10.11, Zwick/Roell, Ulm, Germany). The rationale and the definition of the terms ER and MT, which quantify the elastic (ER) and total energy (MT) absorbed per unit of volume during the bend test until material rupture occurs, have previously been explained.[33,34] Both properties describe energy dissipation effects which may help to prevent undesired fracture scenarios.

Furthermore, to gain additional autonomous information regarding the stability of the mechanical properties of the bis-acryl interim resin materials investigated in the present study, a second independent series of cartridges with the same lot number was placed in the incubator at 37.0 ± 1.0°C for each IRM to simulate storage temperatures higher than 23°C, which may arise unexpectedly during material transport as well as customs and warehouse storage. This elevated temperature representing physiological conditions was deliberately selected to simulate accelerated aging as previously reported[37,38] and to independently verify the results of the storage tests at ambient laboratory conditions. To conduct a simplified accelerated aging test, these cartridges were periodically removed after 3, 6, 9, and 12 months from the incubator to prepare specimens as described above (n = 10), which were likewise stored for 24 hours in distilled water (37.0 ± 1.0°C) prior to material testing. All mechanical properties were determined as mentioned before, but only FS was analyzed and reported to illustrate the results obtained.

Mean values and standard deviations of mechanical properties were calculated, and normality of data distribution was tested by using Kolmogorov-Smirnov and Shapiro-Wilk tests. A three-way MANOVA was performed to test the influence of cartridge storage time, water storage time, and material on the investigated mechanical properties by using the Pillai's trace

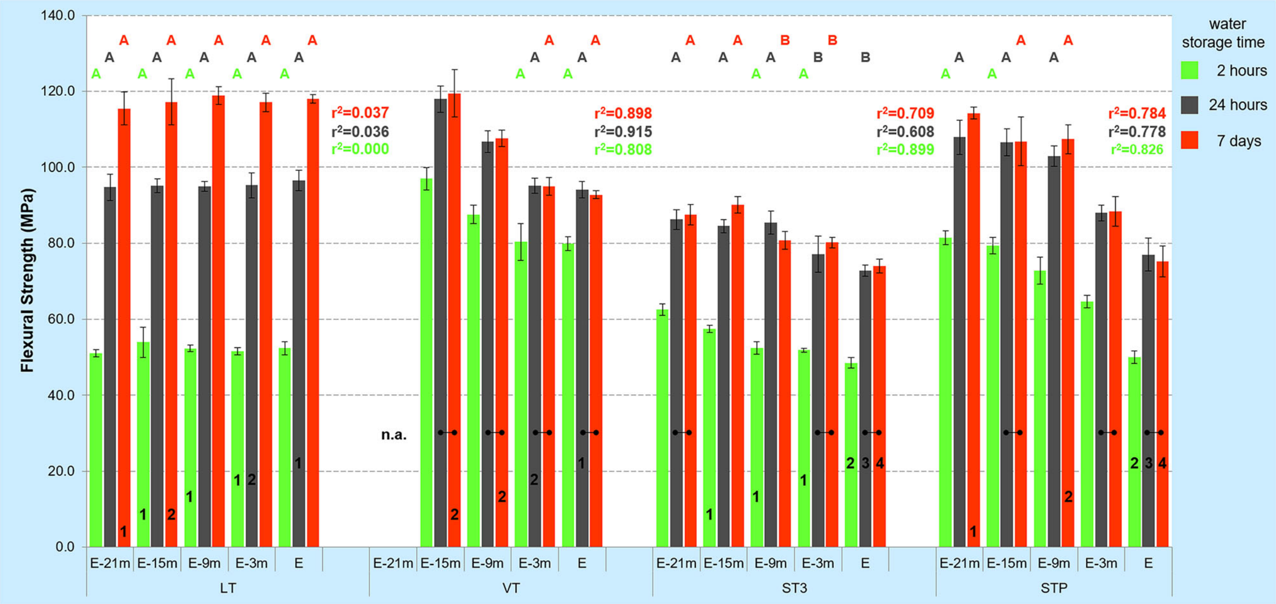

Figure 1 FS results arranged according to the cartridge storage time. Bars show means, whereas indicators represent standard deviations. Identical upper-case letters on the same level represent no significant differences among cartridge storage times for the same IRM and the same water storage time. r^2 values of the linear regression analysis displayed on the right side of each bar group relate to the cartridge storage times of each IRM. Lines with dots connect water storage times without significant differences within one cartridge storage time of the same IRM. Identical numbers on the same level on bars indicate no significant differences among IRM for the same cartridge storage time and the same water storage time ($\alpha = 0.05$). n.a. = not available.

test to account for inhomogeneity of variances. Only the main effects were subsequently analyzed. Data were split according to the respective type of cartridge storage time, water storage time or material, and a series of follow-up one-way ANOVAs were conducted in combination with Welch tests to account for inhomogeneity of variances. Post hoc comparisons were carried out to determine significant differences between groups by using Tukey tests, and Games-Howell tests if variances were not homogeneous. The same statistical tests were used to evaluate the means received for the elevated temperature test at 37°C. Furthermore, to investigate the effect of cartridge storage time on the mechanical properties, in each case a linear regression analysis was applied and r^2 values were calculated. All statistical analyses were performed with SPSS for Windows (version 23.0.0.2, IBM Corp., Armonk, NY) at a significance level of $\alpha = 0.05$. Diagrams were prepared with Excel (Office 16, Microsoft Corp., Redmond, WA).

Results

Table 2 shows the results of the three-way MANOVA. As indicated by the p values, the 3 independent variables "cartridge storage time," "water storage time," and "material," as well as all interactions, showed statistically significant effects. Only the main effects were further analyzed, and the tests of between-subject effects are displayed in Table 3. For the different cartridge storage times, water storage times and materials, the mechanical properties showed significant effects ($p < 0.001$). The calculated means and standard deviations of the investigated mechanical properties for the different test groups are compared in separate bar chart diagrams for FS (Fig 1), ME (Fig 2), MT (Fig 3), and ER (Fig 4).

Luxatemp Plus (LT) showed no significant decrease for any of the mechanical properties when the initial values (E-21m) were compared to those of the last month (E) ($0.054 \leq p \leq 0.871$), irrespective of the water storage time investigated (upper-case letters, Figs 1–4). This outcome was corroborated by the corresponding results acquired for the linear regression analyses ($0.000 \leq r^2 \leq 0.175$).

Visalys Temp (VT) exhibited a progressive degradation of properties with increased cartridge storage time, and significant differences between all means of E-15m and E were found ($0.000 \leq p \leq 0.024$) (upper-case letters, Figs 1–4). This result was supported by high coefficients of determination ($0.275 \leq r^2 \leq 0.915$) and was similarly detected for all 3 water storage times.

For Structur 3 (ST3) and Structur Premium (STP) most mechanical properties showed no alteration during the first 6 months of cartridge storage time (E-15m), whereas with E-9m and higher, a marked degradation of properties was observed for FS and ME, especially for STP ($0.000 \leq p \leq 0.008$). Results for MT and ER appeared to remain unchanged, most obviously for STP and the water storage times of 24 hours and 7 days (upper-case letters, Figs 3 and 4).

The FS results obtained from a separate test series where the cartridges of all IRM were stored at 37°C (Fig 5) showed no significant decrease for LT during an investigation period of

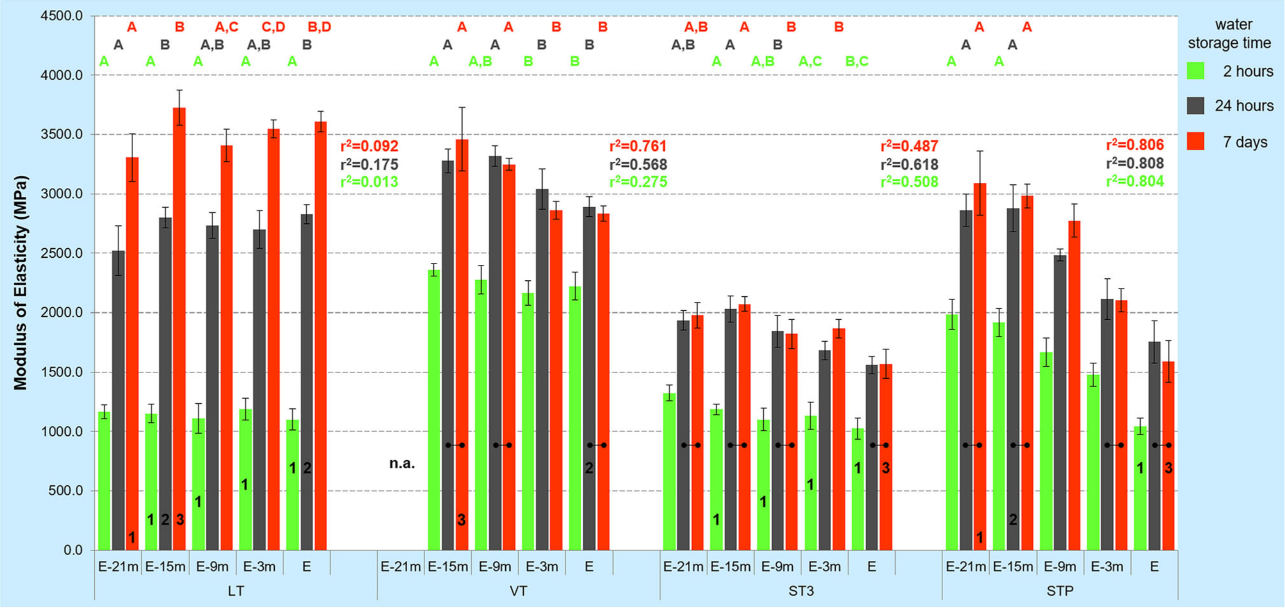

Figure 2 ME results arranged according to the cartridge storage time. Bars show means, whereas indicators represent standard deviations. Identical upper-case letters on the same level represent no significant differences among cartridge storage times for the same IRM and the same water storage time. r^2 values of the linear regression analysis displayed on the right side of each bar group relate to the cartridge storage times of each IRM. Lines with dots connect water storage times without significant differences within one cartridge storage time of the same IRM. Identical numbers on the same level on bars indicate no significant differences among IRM for the same cartridge storage time and the same water storage time ($\alpha = 0.05$). n.a. = not available.

Figure 3 MT results arranged according to the cartridge storage time. Bars show means, whereas indicators represent standard deviations. Identical upper-case letters on the same level represent no significant differences among cartridge storage times for the same IRM and the same water storage time. r^2 values of the linear regression analysis displayed on the right side of each bar group relate to the cartridge storage times of each IRM. Lines with dots connect water storage times without significant differences within one cartridge storage time of the same IRM. Identical numbers on the same level on bars indicate no significant differences among IRM for the same cartridge storage time and the same water storage time ($\alpha = 0.05$). n.a. = not available.

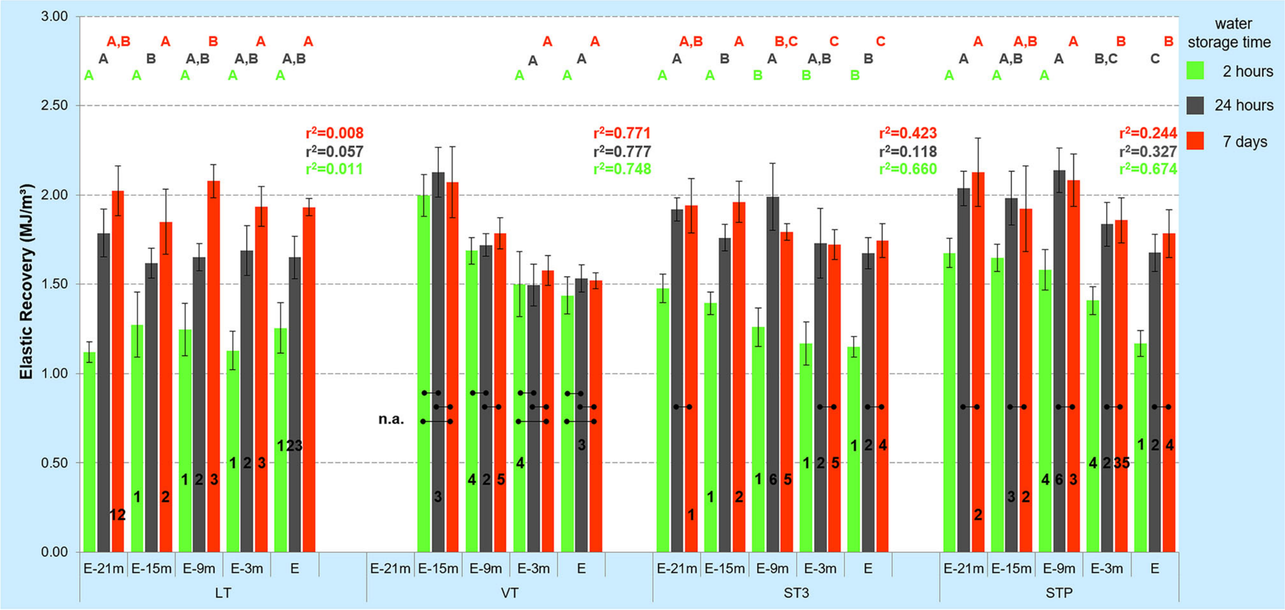

Figure 4 ER results arranged according to the cartridge storage time. Bars show means, whereas indicators represent standard deviations. Identical upper-case letters on the same level represent no significant differences among cartridge storage times for the same IRM and the same water storage time. r^2 values of the linear regression analysis displayed on the right side of each bar group relate to the cartridge storage times of each IRM. Lines with dots connect water storage times without significant differences within one cartridge storage time of the same IRM. Identical numbers on the same level on bars indicate no significant differences among IRM for the same cartridge storage time and the same water storage time ($\alpha = 0.05$). n.a. = not available.

12 months if the means were compared to the initial value at 0m ($0.054 \le p \le 0.815$). Conversely, a progressive decline was observed for VT where the mean of the last month was significantly lower than the initial value ($p < 0.001$). Furthermore, a marked effect was obtained for STP and ST3 where both materials showed premature curing inside of the cartridges after 6 months and 9 months, respectively, which impeded the squeezing out of pastes. The same effect was likewise observed after 4 months incubator time at 37 °C for pastes separated from the cartridges (Fig S6).

Discussion

A major result of the present study is that the IRM tested behaved very differently in terms of the stability of their mechanical properties with increased cartridge storage time and within different water storage times for cured specimens. Markedly different IRM compositions and varying material stabilizations led to premature aging and mechanical properties degradation like that partially observed for VT, STP, and ST3, whereas such destructive effects were not determined for LT. Hence, the null hypothesis, that the mechanical properties are independent of the cartridge storage time, the tested IRM and the water storage time of cured specimens had to be completely rejected.

Previously published investigations that assessed the performance and durability of two-component composite resins unambiguously revealed a strong influence of storage temperature and storage time on the stability of mechanical properties. The findings of several independent studies therefore clearly recommended material storage in a refrigerator to prevent or delay premature aging.[23,25,39,40] Nevertheless, such recommendations seem to be less important today, and many manufacturers of two-part paste systems like luting cements, core build-ups and equally IRM permit the storage of their materials at room temperature (partly up to 28 °C) without explicitly stating the benefit of using a refrigerator. This missing notice was likewise found for all 4 IRM investigated in the present study (Table 1). In fact, for LT and VT there was an explicit note in their user instructions "Do not store in a refrigerator" for as yet unknown reasons,[17,18] while ST3 and STP had a recommended storage temperature range of 4 °C to 23 °C. However, manufacturers of dental restorative materials are obliged to precisely define the shelf life of their products under specified storage and transport conditions for which they guarantee the persistence of the specified characteristics of their devices.[41] In the present study, only LT satisfied this regulation. For all investigated properties, it demonstrated no significant differences between the initial values and the values of the last shelf-life month. Conversely, all other IRM obviously suffered from material changes and possible deterioration processes under the specified storage conditions, and this partially provoked a significant degeneration of properties during the useful life. This finding is of high clinical relevance, as dental practitioners need to rely on the assertions of manufacturers and the stability tests performed. Practitioners do not usually have either the information regarding previous "storage history" of the purchased materials or the reliable methods, know-how and time to assess product specifications to guarantee unconditional quality for their patients.

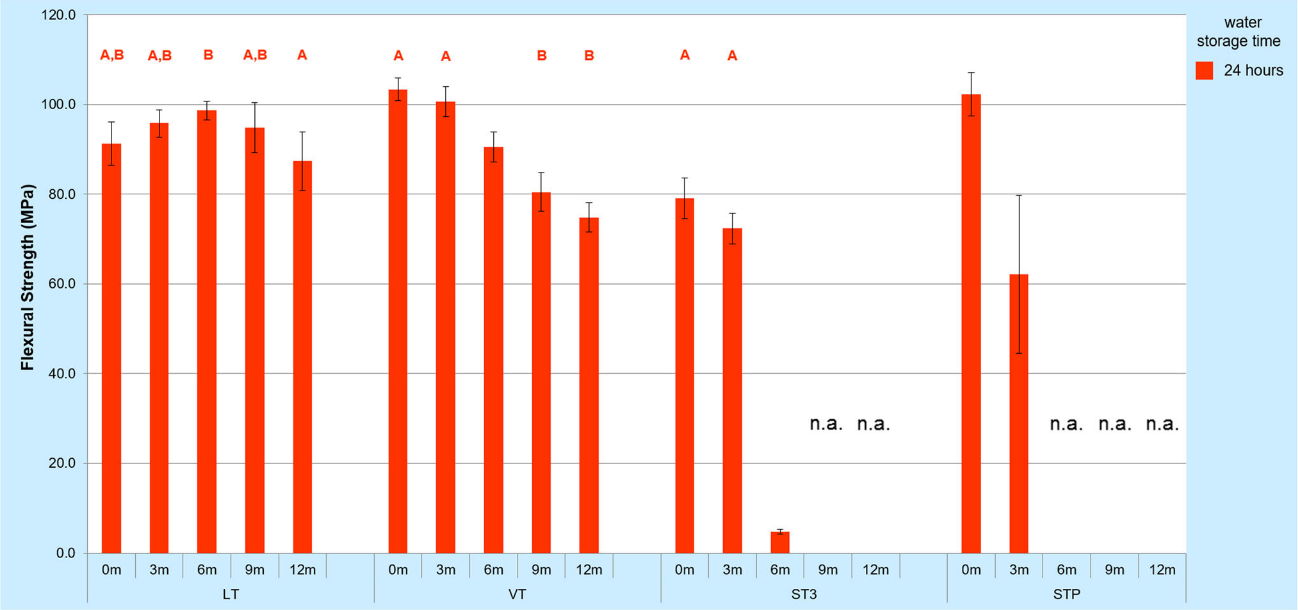

Figure 5 FS results for different cartridge storage times at elevated temperature (37 °C). Bars show means, whereas indicators represent standard deviations. Identical upper-case letters on the same level represent no significant differences among cartridge storage times for one IRM ($\alpha = 0.05$). 0m, 0 months; 3m, 3 months; 6m, 6 months; 9m, 9 months; 12m, 12 months storage time.
n.a. = not available.

No ISO standards that provide exact guidelines for shelf-life testing of dental restorative materials are available. Therefore, the present study compared mechanical properties of IRM in 6-month intervals at well-defined ambient laboratory conditions throughout the entire material shelf life, starting from the date where the cartridges of all selected IRM were disposable. For the storage temperature, the highest permitted common temperature of all IRM was selected, which led to 23°C as specified for ST3 and STP. Since the particular ages of the tested materials, or rather their exact production dates, could not be obtained from the manufacturers, the time plan for the present investigation was related to the expiry dates. This is a possible limitation of the present study together with the unknown storage and transport conditions the investigated materials were exposed to until they were supplied. However, this approach simply reflected clinical practice and all manufacturers asserted a maximum shelf life of 2 years for their materials from the date of filling (Table 1), which for LT, ST3 and STP meant only 3 months of external storage conditions and for VT 9 months. Hence, 15 common months were left for a comparative evaluation of the selected IRM.

Although modern initiator systems,[42–44] other than conventional temperature-sensitive peroxide/amine systems,[45] might be used to cure contemporary IRM, other important factors like specific material composition appear to significantly determine the useful life of IRM cartridges, as demonstrated in this study for LT and VT. Both materials might have similar initiator systems, which was speculatively deduced from identical cartridge systems with a 10:1 mixing ratio (Table 1) usually used for barbituric acid/copper-ion initiations,[43,44] and neither showed any evidence of containing peroxides (Fig S6). Nevertheless, they appeared to have significantly different stability properties showing no deterioration effects for LT during the entire cartridge storage time. This finding is further corroborated by the results of the elevated temperature storage test at 37°C (Fig 5), where again only LT preserved its FS values, obviously attributed to its unique material composition and intrinsic stabilization. Precisely these specific characteristics of LT might likewise be the reason for the unique effect of continuously ascending mechanical properties (FS, ME, ER) observed during the investigation period of 7 days (Figs 1, 2, 4). This finding was previously described for FS and ME[29] as well as for fracture toughness results[8] and was explained with increasing conversion of remaining reactive double bonds and relaxation phenomena within the polymer network.[8] Speculatively, from today's point of view, a consistent increase in crosslink density of the polymer network over time could also likewise explain the effect of increasing mechanical properties. In addition, a former study revealed that even after 7 days of initial curing, post-cure processes readily occurred in composites resulting in improved mechanical properties and degree of conversion.[46] This may help to explain the unique behavior of LT in the present investigation.

Likewise, ST3 and STP appeared to have similar initiator systems, again speculatively deduced from the 1:1 mixing ratio of their cartridges and more apparently inferred from the fact that both IRM evidently contained peroxide (Fig S6). With that, both materials reveal a higher intrinsic risk to suffer from a well-known, temperature-induced degeneration of properties over time, which was extensively described earlier for peroxide systems.[39,40] Exactly this deterioration process was found in the present study and was most obvious for FS and ME values of STP. Both the storage in cartridges over a long time period of 12 months as well as the storage of extruded pastes over a short time of a few months (Figs 5 and S6) at 37°C that induced an accelerated aging process[37,47] even reveal a premature curing effect of respectively one paste of ST3 and STP, which clearly demonstrate the temperature sensitivity and deterioration of properties. Nevertheless, compared with VT, both materials showed only marginal changes regarding ER and MT, especially until the end of their shelf life. This indicates a higher chance to maintain their initial capacity to elastically and plastically dissipate energy,[33] and, from a clinical point of view, to more effectively preserve restoration integrity and resist destructive fracture energy until the end of the shelf life. Such energy dissipation and energy-consuming processes like damping in polymer networks are usually related to plasticizing effects.[48] These are typically substantiated via low glass transition temperatures (T_g)[49] and are held to be associated with water uptake[50] or the presence of residual monomers or short-length oligomers[49,51,52] both acting similarly as plasticizers and consequently reducing T_g. Although T_g and the residual monomer concentrations were not determined in this study their previously well-investigated effect on material properties may help to speculatively explain the results obtained for ST3 and STP as both IRM may have suffered from an ineffective polymerization process, especially in the last months of the shelf life, giving rise to a reduced crosslink density and possibly higher concentrations of residual monomers and oligomers. The influence of this result on long-term restoration stability is not yet known. Unfortunately, meaningful comparative clinical data are still not available to date.

Conclusion

Based on the findings of this in vitro study, the following conclusions were drawn. Increasing cartridge storage times of IRM at ambient laboratory conditions partially induced premature aging with a marked degradation of mechanical properties. While VT, ST3 and STP were significantly affected by this phenomenon, LT showed no significant deterioration of all initial properties during the entire shelf life. For some bis-acryl interim resin materials, the chemical stabilization of its resin and initiator system, as well as the recommended storage conditions, should be revised by the manufacturer to prevent premature aging of the respective materials during shelf life.

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Supporting Information

Additional supporting information may be found online in the Supporting Information section at the end of the article.

Figure S6. Premature curing of ST3 and STP after 4 months incubator at 37 °C.
Figure S7. Explanatory time scale to visualize IRM inspection dates.