INTRODUCTION

In dentistry, cementation is the seating of an indirect restoration on one or several abutment teeth or implants. The primary function of cementation is to provide a reliable retention, a durable seal of the marginal gap and adequate esthetic properties

Different types of cements are available to the clinicians. Conventional cements include zinc phosphate, carboxylate and glass ionomer cements. Although carboxylate and glass ionomer cements establish some degree of chemical adhesion to the tooth structure, the retention of conventionally cemented indirect restorations mainly relies on the frictional forces between the walls of the preparation and the internal walls of the restoration. Thus, a macro-retentive tooth preparation and an excellent marginal fit are required.

Resin composite cements with adhesive properties that can bond to both, dental tissues and the restorative material have further been developed. These minimize invasive dentistry and have become increasingly feasible in restorative dentistry.

Resin composite cements increase the longevity of wing-retained restorations and silica-based ceramic restorations as they decrease the fracture risk and are compulsory for ceramic veneers, inlays and onlays. For restorations of silica-based ceramics, adhesive resin composite cements are indicated to avoid loss of retention, particularly when the restoration lacks optimal retention and resistance form. Their advantages are optimized retention and therefore reduced need for macro-retention, better marginal seal, negligible solubility, and improved esthetic properties when compared to conventional cements. Further, they provide higher compressive, tensile and flexural strength and improved marginal wear resistance.

Resin composite cements can be classified by their adhesive scheme into adhesive and self-adhesive resin composite cements. Adhesive resin composite cements require adhesive tooth surface pre-treatment and are divided into etch and rinse, also called total-etch resin cements and self-etch resin cements depending on the adhesive system (etch and rinse adhesive system or self-etch primers) used prior to cementation. Self-adhesive resin cements do not require any tooth surface pre-treatment. Its incorporated acid-functionalized monomers (predominantly methacrylate monomers with either carboxylic acid groups or phosphoric acid groups) demineralize and infiltrate enamel and dentin. Complete wetting of a rough surface and polymerization lead to micromechanical retention. Simultaneously, acid-base reaction between the acidic functionalities on the monomers and the calcium of hydroxyapatite as well as the alkaline fillers create ionic bonds between tooth and cement and neutralize the initially acidic system. Water is formed during the neutralization reaction, which makes the cement hydrophilic at the beginning. Associated with the neutralization process, the cement converts from a relatively hydrophilic state that wets moist dentin and enamel to a fairly hydrophobic state which limits post-cure swelling and material deterioration.

The setting reaction of resin composite cements can be initiated by self-curing mechanism or light-activation which leads to cross-linking of cement monomers and thus the formation of high molecular-weight polymers.
through free radical polymerization\(^5\),\(^7\). Self-curing resin cements come with a base and a catalyst past. As soon as they are mixed and the added inhibitor is consumed, polymerization starts as a redox reaction. In light-activation, the photo-initiator, most commonly camphorquinone, is activated when exposed to light of a wavelength of 468 nm\(^5\),\(^7\)-\(^9\). It has been shown that light-cured resin cements have a higher conversion rate and better mechanical and chemical stability\(^7\),\(^8\). Many resin composite cements are dual-curing, meaning they offer both activation mechanisms, which allows cement polymerization in areas where light cannot be transmitted in an adequate intensity.

Adhesive resin composite cements show viscoelastic behavior\(^10\). They exhibit a mixture of viscous and elastic properties when undergoing deformation. In very general terms, viscosity is the resistance to flow caused by internal friction between the components in a flowing fluid. In fluids, viscosity is temperature dependent. As temperature rises, intermolecular forces become weaker and viscosity decreases\(^11\),\(^12\). Elasticity is the ability of a material to resist a distorting stress and to regain its original form on removal of the stress. Perfect viscous flow and perfect elastic deformation are the limiting extremes of viscoelastic behavior\(^11\). Rheology describes the viscoelastic behavior of materials. The storage modulus \(G'\) represents the stored deformation energy which becomes a driving force for reforming the structure into its original shape, once the material is released from stress. The loss modulus \(G''\) characterizes the deformation energy that is transformed into heat and therefore lost through internal friction when the material is flowing\(^11\).

Area measurements of cement drops using a drop shape analyzer have recently been used to evaluate the polymerization shrinkage of resin composite cements\(^13\). Hence, contact angle measurements may also provide further insight into the viscous behavior of resin composite cements.

Especially for all ceramic restorations, viscosity must allow the seating of the restoration without excessive pressure in order to avoid stress on the restoration. Further, the polymerization onset defines the processing time, which has to be adequate for a safe clinical handling. The aim of the present study was therefore to analyze viscosity and polymerization kinetics during the setting reaction of six different resin composite cements and how they are influenced by temperature.

**MATERIALS AND METHODS**

Three adhesive (Panavia V5, Kuraray Noritake Dental [PV5]; RelyX Ultimate, 3M Espe [RUL]; Multilink Automix, Ivoclar Vivadent [MLA]) and three self-adhesive (Panavia SA plus, Kuraray Noritake Dental [PSA]; RelyX Unicem 2 Automix, 3M Espe [RUN]; Multilink SpeedCem, Ivoclar Vivadent [MSC]) resin composite cements were chosen to determine their viscous behavior as well as the polymerization rate (Table 1). Viscous behavior was analyzed by a rheological analysis and changes in contact angle of cement drops during polymerization. Polymerization kinetics was assessed by measuring the temperature during polymerization. The working and setting time of the cements as provided by the manufacturers is given in Table 2.

**Rheological analysis**

In a darkroom, a rheological analysis of the six resin cements (Table 1) was performed to determine their elastic and viscous properties (MCR 101, Anton Paar, Graz, Austria). A rotation viscometer with a plate to plate measuring system PP12 with a diameter of 12 mm was used. Measuring distance was set to 0.5 mm. Start delay was 60 s after the cement placement, and measurements were taken at a frequency of 10 Hz at 23°C (logarithmic decrease of deformation \(\gamma=0.25\%\) to 0.001%). The storage modulus \((G')\) representing the elastic behavior and the loss modulus \((G'')\) showing the viscous behavior were plotted over 700 s. Samples were covered during the measurements in order to prevent any light exposure.

**Contact angle**

The change in contact angle (CA) over time of each cement (Table 1) was recorded and the final change of contact angle calculated (Drop Shape Analyzer DSA-100/Software Advance, Krüss, Hamburg, Germany). In order to prevent early cement hardening, a black box was installed around the Drop Shape Analyzer (DSA) and a red-light filter on the light source. Start delay was 60 s after the cement placement on slides and measurements were taken every 20 s at 23°C and 37°C, respectively \((n=5\) for every cement and temperature). For measurements at 37°C the slides were placed on a water circulation-based heating plate (basic immersion circulator, Julabo R5, Julabo, Seelbach, Germany). The left and right CA between cement sample and ceramic surface on both sides (left: L and right: R), CA(L) and CA(R), were evaluated and the mean value CA(M) calculated at every measurement and plotted over 10 min. In case the difference between CA(L) and CA(R) exceeded 10°, the specimen was excluded and the experiment repeated.

**Temperature during polymerization**

Temperature measurements during polymerization of each cement (Table 1) were performed using an infrared thermometer (Optris CT Laser LT, optics: CF2, Optris, Berlin, Germany) in a dark room. Start delay was 60 s after mixing the cement. A drop of the cement was placed on a black paper sheet, and measurements of the heat production were taken \((n=5\) for every cement and temperature) for 16 min and 40 s \((1,000\) measurements). The black paper sheet with the cement sample was placed on a piece of polystyrene for the measurements at 23°C or a heating plate (basic immersion circulator) for the measurements at 37°C. The maximum temperature and its time point were determined based on the mean values of each resin
Table 1  Resin composite cements used in the present study. Information about the composition was taken from safety data sheets and instructions for use provided by the manufacturers.

| Code | Name          | Manufacturer | Type                          | Monomers                                                                                           | Fillers                                                                 | Initiators                  |
|------|---------------|--------------|-------------------------------|---------------------------------------------------------------------------------------------------|------------------------------------------------------------------------|-----------------------------|
| PV5  | Panavia V5    | Kuraray      | Adhesive resin composite cement | Paste A: Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate, Hydrophilic aliphatic dimethacrylate | 38 vol%                                                                 | dl-Camphorquinone           |
|      |               |              |                               | Paste B: Bis-GMA, Hydrophobic aromatic dimethacrylate, Hydrophilic aliphatic dimethacrylate        | - Silanated barium glass filler                                      |                             |
|      |               |              |                               |                                                                                                    | - Silanated fluoroalminosilicate glass filler                        |                             |
|      |               |              |                               |                                                                                                    | - Colloidal silica                                                   |                             |
|      |               |              |                               |                                                                                                    | - Silanated aluminium oxide filler                                   |                             |
|      |               |              |                               |                                                                                                    | Particle size: 0.01–12 µm                                            |                             |
| PSA  | Panavia SA plus | Kuraray       | Self-adhesive resin composite cement | Paste A: 10-MDP, Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate, HEMA | 40 vol%                                                                 | dl-Camphorquinone           |
|      |               |              |                               | Paste B: Hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate                  | - Silanated barium glass filler                                      |                             |
|      |               |              |                               |                                                                                                    | - Silanated colloidal silica                                        |                             |
|      |               |              |                               |                                                                                                    | Particle size: 0.02–20 µm                                            |                             |
| RUL  | RelyX Ultimate | 3M Espe      | Self-adhesive resin composite cement | Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers | 43 vol%                                                                 | Sodium toluene-4-sulphinate, |
|      |               |              |                               | Catalyst paste: methacrylate monomers                                                                   | - Silanated fillers                                                   | Disodium peroxodisulphate,  |
|      |               |              |                               |                                                                                                    | - Alkaline (basic) fillers                                           | Tert-butyl 3,5,5-trimethylperoxyhexanoate |
|      |               |              |                               |                                                                                                    | Particle size: 13 µm                                                  |                             |
| RUN  | RelyX Unicem 2 Automix | 3M Espe | Self-adhesive resin composite cement | Base paste: phosphoric acid modified methacrylate monomers, bi-functional methacrylate | 43 vol%                                                                 | Sodium toluene-4-sulphinate, |
|      |               |              |                               | Catalyst paste: methacrylate monomers                                                                    | - Silanated fillers                                                   | Persulfate, Tert-butyl 3,5,5- |
|      |               |              |                               |                                                                                                    | - Alkaline (basic) fillers                                           | trimethylperoxyhexanoate    |
|      |               |              |                               |                                                                                                    | Particle size: 12.5 µm                                                 |                             |
| MLA  | Multilink Automix | Ivoclar Vivadent | Adhesive resin composite cement | Base paste: Bis-GMA, HEMA, 2-dimethylaminooethyl methacrylate | 40 vol%                                                                 | Dibenzoyl peroxide          |
|      |               |              |                               | Catalyst paste: ethoxylated bisphenol A dimethacrylate, UDMA, HEMA                                             | - Barium glass                                                        |                             |
|      |               |              |                               |                                                                                                    | - Ytterbium trifluoride                                               |                             |
|      |               |              |                               |                                                                                                    | - Spheroid mixed oxide                                                |                             |
|      |               |              |                               |                                                                                                    | Particle size: 0.25–3.0 µm                                             |                             |
| MSC  | Multilink SpeedCem | Ivoclar Vivadent | Self-adhesive resin composite cement | Base paste: UDMA, TEGDMA, polyethylene glycol dimethacrylate | 40 vol%                                                                 | Dibenzoyl peroxide          |
|      |               |              |                               | Catalyst paste: polyethylene glycol dimethacrylate, TEGDMA, Methacrylated phosphoric acid ester, UDMA | - Barium glass                                                        |                             |
|      |               |              |                               |                                                                                                    | - Ytterbium trifluoride                                               |                             |
|      |               |              |                               |                                                                                                    | - Silicium oxide                                                       |                             |
|      |               |              |                               |                                                                                                    | Particle size: 0.1–7 µm                                                 |                             |

10-MDP: 10-Methacryloyloxydecyl dihydrogen phosphate, Bis-GMA: bisphenol A-glycidyl methacrylate, HEMA: 2-hydroxyethyl methacrylate, UDMA: urethane dimethacrylate, TEGDMA: triethylene glycol dimethacrylate
Table 2  Working and setting time of the resin composite cements PV5, PSA, RUL, RUN, MLA and MSC according to the manufacturer’s specifications

| Cement | Working time | Setting time |
|--------|--------------|--------------|
| PV5    | At 23°C (after initial dispersing): 2 min  
At 37°C (after coming into contact with Tooth Primer): 60 s | At 37°C (after placement of the restoration): 3 min (self-cure) |
| PSA    | At 23°C (after initial dispersing): 1 min  
At 37°C (after insertion into the oral cavity): 40 s | At 23°C (after placement of the restoration): 12 min (self-cure)  
At 37°C (after placement of the restoration): 5 min (self-cure) |
| RUL    | From start of mixing: 2.30 min | After start of mixing: 6 min |
| RUN    | From start of mixing: 2.30 min | After start of mixing: 6 min |
| MLA    | At 23±1°C: approx. 3 min  
At 37±1°C (in combination with Multilink Primer): approx. 2 min | At 23±1°C, including working time: approx. 8 min  
At 37±1°C (in combination with Multilink Primer), including working time: approx. 5 min |
| MSC    | At 23±1°C: 150±30 s  
At 37±1°C: 110±30 s | At 23±1°C (without working time): 240±60 s  
At 37±1°C (without working time): 160±40 s |

Fig. 1  Rheological data of resin composite cements PV5, PSA, RUL, RUN, MLA and MSC. G': storage modulus, G'': loss modulus.

Fig. 2  Relative alteration of contact angle (CA) as a function of time for every examined resin composite cement PV5, PSA, RUL, RUN, MLA and MSC (mean value of n=5 for every cement). A) at 23°C, B) at 37°C

Statistics

Maximum temperature values were analyzed for normal distribution using Shapiro-Wilk test and then compared with one-way ANOVA (p<0.05) followed by post-hoc Fisher LSD test (p<0.05).

RESULTS

Rheological analysis

The plotted storage modulus (G') representing the elastic behavior and the loss modulus (G'') revealing the viscous behavior of each cement over 700 s are displayed in Fig. 1. Cement MSC showed the earliest and comparably fastest increase in viscosity. MLA displayed a higher viscosity at the beginning that proceeded however slowly and did not reach a plateau until after 700 s. PV5 initially had a higher flowability with a sudden and fast increase in viscosity in comparison to
the other cements. PSA, RUL and RUN showed a similar curve progression. After a constant initial phase, that is shorter than for PV5, viscosity rose markedly but less fast compared to MSC and PV5.

Contact angle
The relative change in CA at 23°C and 37°C is presented in Fig. 2. The viscosity of RUL, RUN and MLA increased at 37°C compared to 23°C whereas it decreased for PV5, PSA and MSC. RUL displayed the highest relative alteration of CA over time at 23°C and PV5 at 37°C. All cements had initially a faster relative alteration of CA at 37°C and reached the final angle earlier than at 23°C.

Final change of the CA was calculated in % and is displayed in Fig. 3 illustrating again the increase in viscosity of PV5, RUL, RUN and MLA compared to the decrease of PSA and MSC when raising the temperature from 23°C to 37°C. The highest final change in contact angle at 23°C was recorded for RUL and at 37°C for PV5. Especially for RUL final change in contact angle at 23°C was twice as high as at 37°C. MLA showed the lowest final change in contact angle overall both at 23°C and at 37°C.

Temperature during polymerization
The plotted temperature of the cements during polymerization at 23°C and 37°C is displayed in Fig. 4. In general, temperature peaks were more pronounced and at earlier time points at 37°C than at 23°C. MSC

![Fig. 3 Final change in contact angle (CA) for the examined resin composite cements PV5, PSA, RUL, RUN, MLA and MSC at 23°C and at 37 °C (mean values and standard deviation)](image)

![Fig. 4 Temperature profile during polymerization for the examined resin composite cements PV5, PSA, RUL, RUN, MLA and MSC (mean value of n=5). A) at 23°C, B) at 37°C](image)

| cement | Tmax [°C] | TP [s] | Tmax [°C] | TP [s] |
|--------|-----------|--------|-----------|--------|
| PV5    | 25.3 (±0.3) | 665 (±41) | 44.5 (±1.0) | 164 (±10) |
| PSA    | 22.5 (±0.3) | 697 (±70) | 38.2 (±0.2) | 207 (±61) |
| RUL    | 23.1 (±0.2) | 767 (±49) | 39.4 (±0.3) | 189 (±13) |
| RUN    | 23.4 (±0.6) | 637 (±40) | 40.0 (±0.5) | 160 (±9) |
| MLA    | 25.6 (±0.3) | 789 (±12) | 41.0 (±0.8) | 299 (±11) |
| MSC    | 22.8 (±0.3) | 427 (±84) | 40.1 (±0.3) | 120 (±12) |

Table 3 Maximum temperature (Tmax) for the resin composite cements PV5, PSA, RUL, RUN, MLA and MSC while polymerizing at 23°C or 37°C respectively with the corresponding time points (TP) after cement mixture (mean and standard deviations).

Statistically significant differences between peak temperatures of the cements are marked with varying superscript letters (vertical comparison at 23°C uppercase letters, 37°C lowercase letters, p<0.05).
revealed the earliest peak at room temperature and at 37°C. For MLA the temperature peak was comparatively late at 23°C as well as at 37°C. At 37°C, temperature of MLA increased slowly and steadily until the maximum temperature of 40.9°C was reached. PV5 showed a very pronounced and slim peak at 37°C. PSA, RUL and RUN did not reveal very clear temperature peaks at 23°C. At 37°C RUL and RUN showed similar temperature peaks whereas the PSA peak was less pronounced. The corresponding maximum temperature values for the peaks with statistical comparison between the cements are given in Table 3.

**DISCUSSION**

The results demonstrated strong differences in viscous behavior of the different resin composite cements during their setting reaction. In a clinical situation, the cements are mixed at room temperature and applied to the restoration, which is subsequently placed in the oral cavity. During mixing and application, the viscosity should be low in order to allow proper seating of the restoration and to have sufficient working time. Immediately after placement in the mouth the viscosity should increase to avoid cement residues in the surrounding soft tissue. Thus, polymerization should only start after a sufficient working time, which also permits the removal of excess cement, but should then progress in due time.

The obtained results demonstrate that for all cements temperature is higher during the polymerization reaction. At 37°C, the temperature peak is earlier, more pronounced and temperature increased when compared to 23°C. Higher temperatures also have an impact on viscosity. In fluids it results in more flexible molecular networks and thus lower viscosity. As the polymerization reaction proceeds, the polymer network becomes more rigid and viscosity increases. In this study, RUL, RUN and MLA increase in viscosity as temperature rises. It is obvious, that the influence of temperature on the acceleration of the polymerization reaction outweighs the impact that temperature has on viscosity. In contrast, PV5, PSA and MSC decrease in viscosity during temperature increase. In this case the cements become more fluid and the influence of polymerization reaction on viscosity overweighs only later.

Rheological and temperature measurements correspond in terms of curve progression. MSC shows an early rise with an early peak in the temperature figure and an early bend in the rheological analysis. After an initially constant phase, PV5 displays a sudden and steep ascent in both rheology (Fig. 1) and temperature (Fig. 4). The MLA curve rises rather slowly and steadily in both characteristics and shows a late peak in the temperature profile. PSA, RUL and RUN show similar peaks in the temperature profile and similar bends in the rheological analysis.

Neither the rheological analysis nor the temperature measurement correlates with the CA-measurement over time. Multiple factors such as cement viscosity, polymerization, shrinkage or surface wetting influence the change in CA over time and therefore make it difficult to draw conclusions. Measuring the change in CA over time might not be an appropriate method to examine viscous behavior of resin cements due to the multiple parameters influencing the results. Rheological analysis was performed at 23°C only since there was no possibility to heat the rheometer plate up to 37°C.

It can be seen, that generally at 23°C viscosity starts to increase before temperature rises (Figs. 1 and 4A). Ideally, viscosity should increase at the end of working time, when the restoration is placed correctly. For PV5 viscosity starts to increase after ~6.1 min at 23°C (Fig. 1), which is sufficient time to place the restoration as the manufacturer indicates 2 min working time at 23°C (Table 2). At 37°C the temperature rises after around 2.2 min for PV5 (Fig. 4). Viscosity of PSA starts to increase after ~3.6 min at 23°C (Fig. 1) and temperature starts to rise after ~1.7 min at 37°C (Fig. 4), which corresponds to the manufacturer’s specifications of 1 min at 23°C and 40 s after insertion into the oral cavity (Table 2). For RUL and RUN viscosity starts to rise after ~5.2 min and ~5.5 min respectively at 23°C (Fig. 1) and temperature at 37°C after ~3.2 min and ~ 2.9 min (Fig. 4), which is also in accordance with the manufacturer’s specifications of 2.5 min for both cements (Table 2). Viscosity of MLA starts to rise after ~4.5 min and is already slightly rising before (Fig. 1) at 23°C. At 37°C temperature is already on the rise after 60 s (start of the measurements) (Fig.4). This implies that the manufacturer’s working time specifications of 3 min at 23°C and 2 min at 37°C are too short (Table 2). For MSC viscosity rises after 1.6 min at 23°C (Fig. 1) and temperature rises strongly after 60 s at 37°C (Fig. 4) which implies again that the manufacturer’s specifications of 150±30 s at 23°C and 110±30 s at 37°C, are too short (Table 2).

For the interpretation of the maximum temperature of each cement it must be considered that the mass of the cement used in the present investigation was quite high compared to the mass used for cementation of a restoration where cements are applied in thin layers only. Therefore, it would be unjustified to deduce any risk of a pulpal damage from the temperature increase observed in the present study.

It can be stated that Kuraray cements (PV5, PSA) become less viscous at 37°C and then polymerize quickly. 3M Espe cements (RUL, RUN) start with low viscosity at 23°C. At 37°C viscosity rises, and polymerization takes place quickly. Ivoclar Vivadent cements (MLA, MSC) do not show uniform behavior. Both start with a high viscosity. MSC polymerizes very fast and MLA rather slowly and steadily. Each manufacturer follows his own philosophy and the clinician has a range of materials to choose the one, which fits best to his own expectation and practice.

**CONCLUSIONS**

Within the limitations of this *in vitro* study, the following conclusions were drawn:
1. Due to differing polymerization kinetics different composite resin cements demonstrate strong variations in their viscous behavior during their setting reaction.
2. PV5 shows excellent viscous behavior in respect to working time and setting reaction.
3. Measuring the change in contact angle over time seems not to be an appropriate method to examine viscous behavior of resin cements as multiple interplaying factors influence the outcome.

ACKNOWLEDGMENTS

The authors do not have any financial interest in the companies whose products are included in this article. The laboratory support of Fredy Schmidli is acknowledged.

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