Development of novel measurement method for consistency of resin cements

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The aim of this study was to develop a new method to evaluate the consistency of resin cements by modeling the operation of setting a crown. First, the weight of the resin cements and the inner surface area of the crown were measured to configure the conditions for consistency evaluation using a model abutment tooth and a model crown. As the evaluation method, a 200 mm² contact surface area of a glass plate was placed on top of 30.0 mg of resin cement paste, and a pressure of 10.0 N was applied to the glass plate. When the paste was spread over the entire bottom surface of the glass plate, it was designated a “Pass”. A “Pass” result was obtained for all resin cements tested and a “Fail” result for all resin composites, indicating the usefulness of the new method for assessing cement flowability when setting a crown.

Keywords: Resin cements, Consistency, Evaluation method, Scanning electron microscopy, Adhesive dentistry

INTRODUCTION

Resin cements are one type of dental cement used for the luting of fixed dental prostheses, and are widely used in clinical practice. The component compounds and composition of resin cements have been improved to prevent fracture and displacement of fixed dental prostheses, and their functions have diversified according to clinical needs. Many studies have been reported on the bonding effectiveness of resin cementsi-6. Sarr et al. compared the bonding effectiveness of nine resin cements used to lute ceramic to dentin, and reported that a sufficient immediate bond strength could be obtained, depending on the resin cement, even with self-adhesive resin cements that do not use a separate dental adhesive (i.e. simple use)7. Mechanical properties, such as the compressive strength and flexural strengths of resin cements have also been often studied. Piwowarczyk and Lauer studied the compressive and flexural strengths of twelve luting cements from different material classes, i.e., resin cements, resin-modified glass ionomer cements, glass ionomer cements, and zinc phosphate cements. They reported that generally the highest measured compressive and flexural strengths were achieved by resin cements, after which came self-adhesive resin cements. Kim et al. compared the compressive strength, diametral tensile strength, and microhardness of six self-adhesive resin cements using different activation modes (self-cured and light-cured) and testing time (immediately, 24 h, and thermocycling), and concluded that, within the limitations of the study, all the cements demonstrated clinically suitable strength values.

In the clinical use of resin cements, the exact placement of the crown with a abutment tooth is crucial issue; therefore, it is important to also consider the handling properties of resin cements when evaluating their characteristics. The handling properties include film thickness, setting time, working time, sensitivity to ambient light, and consistency, the latter of which is an important characteristic because the essential purpose of the cement is to completely fill the required space. In the evaluation of handling properties, it is thus also necessary to evaluate if the resin cement has appropriate consistency for successful setting, so that the resin cement can be evenly spread in the space between the prosthetic appliance and the abutment tooth, and that excess cement can flow out. In response to the increasing variation in types of resin cement, “ISO 4049: 2019 Dentistry —Polymer-based restorative materials”7,7 and “ISO/TC 16506: 2017 Dentistry —Polymer-based luting materials containing adhesive components”7,7 have been established as specifications for various properties of resin cement. However, there is no specification for consistency as an evaluation of the handling properties.

Regarding a method for consistency measurement, there is only a standard defined for zinc phosphate cement8. In this method, 0.500 mL of mixing cement is placed on a smooth glass plate, on top of which a 0.02 kg glass plate and a 0.10 kg weight are placed, and the diameter of the cement spread is measured. This method, and modifications of it, could be used to quantify the consistency of resin cement; however, only a few studies have been reported9,10. Although it is possible to compare the flowability of a resin cement from the results obtained by this method, it is not possible to determine whether the consistency is suitable for setting a crown on an abutment tooth. The purpose of the present study was to develop a new method to evaluate the consistency of resin cements by modeling the operation of setting a crown with resin cements. The conditions for the novel
Materials and methods were configured using a model abutment tooth and a model crown that would be attached. The validity of this method was investigated by evaluating the consistency of commercially available resin cements.

### Materials and Methods

**Measurement of the weight of the cement used to set the crown**

A model of a mandibular second molar formed for a jacket crown (A55A-461, Nissin, Kyoto, Japan) was used as a model abutment tooth. A crown made from a resin composite block (Katana Avencia P block, Kuraray Noritake Dental, Tokyo, Japan) was used as a model crown. The crown was fabricated by taking an optical impression of the model abutment tooth using a scanner (D850, 3Shape, Copenhagen, Denmark), designing the crown by computer-aided design (CAD; DENTAL SYSTEM, 3Shape) based on the digital impression data, creating the machining data by computer-aided manufacturing (CAM; hyperDENT, FOLLOW-ME Technology, Munich, Germany), and milling the resin composite block (Katana Avencia P block, Kuraray Noritake Dental) in a milling machine (DWX-52DC, DGSHAPE, Shizuoka, Japan). PANAVIA SA Cement Plus Automix (PC; Kuraray Noritake Dental) was used as a luting cement (Table 1), and the crown was set on the abutment tooth with finger pressure. The excess cement was removed after being semi-cured by irradiation for a short time with a dental curing unit (PENCURE 2000, J. Morita. Mfg., Kyoto, Japan) and the cement was cured by light irradiation for 10 s on each side using the dental curing unit.

The following procedure was employed to measure the weight of cement used to set the crown. First, after the crown was fabricated and before it was set on the abutment tooth, the crown and the abutment tooth were weighed together (m1) using an electronic balance (Mettler Toledo, Columbus, OH, USA). After the crown was set on the abutment tooth, the weight of the entire specimen (**i.e. the abutment and crown**) was measured (m2). The difference between m2 and m1 was determined to be the actual weight of the cement used to set the crown. The cement weight was measured for four specimens and the mean value was determined.

**Measurement of the inner surface area of the crown**

The specimen was cut along the long axis of the buccolingual face to measure the thickness of the cement layer between the crown and the abutment tooth (Fig. 1b). The cut surface was observed using scanning electron microscopy (SEM; SU3500, Hitachi, Tokyo, Japan) and the thicknesses of the cement layer at the occlusal plane (3 point) and the central part of the axial plane (3 point) were measured from the SEM images. The volume of the cement used to set the crown was obtained from the weight of the resin cement determined in the previous section and the density of PC. The area calculated by dividing the volume of the resin cement by the thickness of the cement layer was the inner surface area of the crown.

**Consistency evaluation method**

Figure 2 shows a schematic diagram of the novel method to evaluate consistency. Glass plate 1 is the base on which

| Material                              | Manufacturer | Lot No. | Composition                                                                 |
|---------------------------------------|--------------|---------|-----------------------------------------------------------------------------|
| PANAVIA SA Cement Plus Automix         | Kuraray Noritake Dental | 000268 | Paste A: MDP, Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, HEMA, silanated barium glass filler, silanated colloidal silica, CQ, peroxide, catalysts, pigments |
|                                       |              |         | Paste B: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, silanated barium glass filler, surface treated sodium fluoride, accelerators, pigments |
|                                       |              |         | The total amount of inorganic filler is ca. 40 vol%. The particle size of inorganic fillers ranges from 0.02 to 20 μm. |
| iCEM Self Adhesive                    | Kulzer       | KO10050 | Acidified urethane, di-, tri-, and multifunctional acrylate monomers, 49% filler by weight of sub-micron and micron sized particles |
| BisCem                                | BISCO        | 1800006535 | Base: Bis-GMA, proprietary, glass filler, Catalyst: bis[2-(methacryloyloxy)ethyl] phosphate, HEMA, bis(glyceryl 1,3 dimethacrylate)phosphate, dibenzoyl peroxide, glass fillers |
|                                       |              |         | Glass fillers have particle sizes from 0.02 to 5.0 μm (average). |
| CLEARFIL AP-X                         | Kuraray Noritake Dental | 000097 | Bis-GMA, TEGDMA, silanated barium glass filler, silanated silica filler, silanated colloidal silica, CQ, catalysts, accelerators, pigments |
|                                       |              |         | Micro-hybrid, 85% fillers (by weight) of silanated barium glass filler, silanated silica filler, and silanated colloidal silica |

MDP: 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA: bisphenol A diglycidylmethacrylate; TEGDMA: triethyleneglycol dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; CQ: DL-camphorquinone.
the specimen is placed and it has a flat and sufficient area. The amount of resin cement to be used in the test was configured based on the weight of the cement used to set the crown. Glass plate 2 corresponds to the crown to be used, and its area was configured based on the inner surface area of the crown. Resin cement paste (30.0 mg) was placed at the center of flat glass plate 1. The flat and uniformly thick glass plate 2 with a contact surface area of 200 mm² and a thickness of 5.00 mm was then placed on top of the resin cement paste. A 10.0 N load was subsequently applied to the center of the top surface of glass plate 2 for 1.00 min, after which the spread of the resin cement was observed. If the paste was spread over the entire bottom surface of glass plate 2, which is considered to be a crown, it was designated a “Pass”, and if it was not spread over the entire surface, it was designated as a “Fail”. The measurement was performed three times (n=3) for each product.

Evaluation of consistency of commercial resin cements
Three commercially available dual-cure type self-adhesive resin cements, PANAVIA SA Cement Plus Automix (PC; Kuraray Noritake Dental), iCEM Self Adhesive (IC; Kulzer, Hanau, Germany), and BisCem (BC; Bisco, Schaumburg, IL, USA), were tested using the developed method to evaluate consistency (Fig. 2). CLEARFIL AP-X (AP; Kuraray Noritake Dental), a universal type resin composite for filling, was used as a comparison material. Table 1 gives the manufacturer specifications for each cement.

RESULTS

Weight of resin cement and the inner surface area of the crown
The mean weight of the resin cements between the crown and the abutment tooth was 30.0 mg (S.D. 1.60 mg, n=4). Based on the measured weight and the density of PC (1.80×10³ kg/m³), the volume of resin cements used to set the crown was 16.0 mm³. Measurement of the cement layer thicknesses on the occlusal plane and the axial plane at three points in the SEM images for each plane (Fig. 3) indicated that the mean thickness of the former was 80.0 μm (n=3) and that of the latter was 120 μm (n=3), and the total mean thickness of the cement layer was ca. 100 μm (n=6). The volume of resin cement divided by the thickness of the cement layer can be regarded as the inner surface area of the crown, which was calculated to be 160 mm².

Evaluation of consistency of commercial resin cements
The new test to evaluate consistency was conducted...

![Fig. 1](image1.png)

Test specimens consisting of a model abutment tooth and a model crown.
(a) Test specimen: a model abutment tooth cemented with a crown. (b) Sectioned specimen along the long axis of the buccolingual face for measurement of the cement layer thickness.

![Fig. 2](image2.png)

Schematic diagram of the setup for consistency measurement.

![Fig. 3](image3.png)

Representative scanning electron microscopy micrograph of the cement layer of a model with a crown cemented to an abutment tooth with resin cement. The average thickness of the cement layer between the crown and the abutment tooth was observed to be ca. 100 μm. AT: abutment tooth; Cr: crown; RC: resin cement.
Table 2  Results of consistency tests

|                  | 1st | 2nd | 3rd |
|------------------|-----|-----|-----|
| PANAVIA SA Cement Plus Automix | Pass | Pass | Pass |
| iCEM Self Adhesive | Pass | Pass | Pass |
| BisCem           | Pass | Pass | Pass |
| CLEARFIL AP-X    | Fail | Fail | Fail |

Fig. 4  Photographs of pastes after 10.0 N loading for 1.00 min.
(a) PC (resin cement) spread over the entire surface of glass plate 2 with excess cement flowing out. “Pass”. (b) IC (resin cement) with the same spread as PC. “Pass”. (c) BC (resin cement) spread over the entire surface of glass plate 2, although the excess cement flowing out is less than PC and IC. (The thickness of glass plate 2 hides parts of the excess cement flow-out area.). “Pass”. (d) AP (high-viscosity composite resin for filling), where the paste does not spread over the entire surface of glass plate 2. “Fail”. Black arrows: paste spread over the entire surface of glass plate 2. White arrows: paste not spread to the corners of glass plate 2.

DISCUSSION

The thickness of the cement layer with the model crown on the model abutment tooth was determined to be ca. 100 μm in the present study. Katoh et al. studied the influence of abutment taper and cement space on the gap size between the crown and abutment tooth in the setting of CAD/CAM crowns with resin composite blocks, using cements with different consistency under a 147 N load. The results indicated gap sizes in the range of 5.00 to 460 μm, depending on conditions and sites\(^\text{13)}\). The thicknesses of the cement layer measured in the present study were between 80.0 and 120 μm, which were within the range reported by Katoh et al.\(^\text{13)}\). Owitayakul et al. investigated setting zirconia crown models on model abutment teeth with two different types of resin cements under 50.0 N load, and the cement layer thicknesses were measured to have mean values in the range of 28.0 to 208 μm\(^\text{14)}\). In the present study, the crown was set using finger pressure, so that a cement thickness of ca. 100 μm seems to be reasonable.

The conditions for the proposed cement consistency test were as follows. The amount of resin cement was determined from the difference between \(m_1\) and \(m_2\), and was set to 30.0 mg. The lower area of glass plate 2 corresponds to the inside area of the crown, i.e., it should be 160 mm\(^2\). In this study, a glass plate with an area of 200 mm\(^2\), as used in the measurement of film thickness according to ISO 4049: 2019 and ISO/TS 16506: 2017, was used for compatibility with the existing apparatus for the evaluation of resin cement. This difference in area was deemed acceptable for the purposes of the test, as it would lead to a stricter evaluation. The load is the force that is applied when the crown is set on the abutment tooth. Zortuk et al. measured the load while setting of crowns by 15 dentists (9 males and 6 females) using model crowns and abutment teeth, and the minimum and maximum loads were reported to be 20.5 and 61.8 N, respectively\(^\text{15)}\). Considering that resin cement is used by a large number of dentists in clinical
practice, it must be able to spread out into the cement space even under a small load; therefore, a load lower than 20.5 N was selected to reflect clinical practice, and 10.0 N was adopted.

Three commercial resin cement products (PC, IC, and BC) were measured under conditions of 30.0 mg of resin cement, 200 mm² cement contact area, and 10.0 N load. All the specimens spread over the entire surface of plate 2 and this was considered as a “Pass” (Figs. 4a–c). In contrast, AP, a high-viscosity resin composite for filling, did not spread over the entire surface of glass plate 2, and was thus considered as a “Fail” (Fig. 4d). In some clinical cases, a high-viscosity resin composite for filling has been used as a luting material for the setting of aesthetic prosthetic appliances. However, the viscosity at room temperature is too high for luting, and preheating to reduce the viscosity for setting has been reported, which corresponds to the study in this comparison experiment 16). The test was conducted with three specimens, and the results were in good agreement with each other. Within each group, there were few differences between the results for the test samples, indicating that the viscosity of the paste was the most important factor, and not subsequent processing conditions. Unlike bond strength and mechanical strength tests, there was no molding, curing and/or storage processes in the present test, and this may affect the normality of the results. Therefore, it is considered sufficient to conduct the evaluation only three times.

This study proposed a new test method to evaluate the consistency of resin cements. We consider that it is preferable to increase the number of commercially available product tests to verify the usefulness of this consistency evaluation method. In addition, the clinical handling properties of the resin cement should not be determined by this method alone, but should be comprehensively evaluated in combination with the results of other tests such as film thickness and curing time. The results of this study indicate that the consistency evaluation method using this model has potential as a method to determine whether a resin cement to be tested has appropriate flowability for the setting of prosthetic appliances.

CONCLUSION

The new method to evaluate consistency developed in this study gave a “Pass” result for all resin cements and a “Fail” result for the resin composite. This indicates that the developed method can be used to determine whether a resin cement has appropriate flowability for setting a crown.

REFERENCES

1) Sarr M, Mine A, De Munck J, Cardoso MV, Kane AW, Vreven J, et al. Immediate bonding effectiveness of contemporary composite cements to dentin. Clin Oral Investig 2010; 14: 569-577.
2) Schittity E, Goff SL, Besnault C, Sadoun M, Ruse ND. Effect of water storage on the flexural strength of four self-frequency adhesive resin cements and on the dentin-titanium shear bond strength mediated by them. Oper Dent 2014; 39: 171-177.
3) De Munck J, Vargas M, Van Landuyt K, Hikita K, Lambrechts P, Van Meerbeek B. Bonding of an auto-adhesive luting material to enamel and dentin. Dent Mater 2004; 20: 963-971.
4) Lin J, Shinya A, Gomi H, Shinya A. Bonding of self-adhesive resin cements to enamel using different surface treatments: bond strength and etching pattern evaluations. Dent Mater J 2010; 29: 425-432.
5) Piwowarczyk A, Lauer HC. Mechanical properties of luting cements after water storage. Oper Dent 2003; 28: 535-542.
6) Kim AR, Jeon YC, Jeong CM, Yun MJ, Choi JW, Kwon YH, et al. Effect of activation modes on the compressive strength, diurnal tensile strength and microhardness of dual-cured self-adhesive resin cements. Dent Mater J 2016; 35: 298-308.
7) ISO 4049: 2019. Dentistry, Polymer-based restorative materials, the International Organization for Standardization. Geneva: Switzerland; 2019.
8) ISO/TS 16506: 2017. Dentistry, Polymer-based luting materials containing adhesive components, the International Organization for Standardization. Geneva: Switzerland; 2017.
9) American Dental Association Specification No. 8 for Zinc Phosphate Cement. American National Standard Z156.8 1970; 187-191.
10) Van Meerbeek B, Inokoshi S, Davidson CL, De Gee AJ, Lambrechts P, Braem M, et al. Dual cure luting composites Part II: Clinically related properties. J Oral Rehabil 1994; 21: 57-66.
11) Fraga RC, Luca-Fraga LRL, Pimenta LAF. Physical properties of resinous cements: an in vitro study. J Oral Rehabil 2000; 27: 1064-1067.
12) Bagheri R. Film thickness and flow properties of resin-based cements at different temperatures. J Dent (Shiraz, Islamic Repub. Iran) 2013; 14: 57-63.
13) Katoh H, Kasahara S, Kimura K, Okuno O. Effect of taper, cement space and consistency of luting agents on the fitness of crowns fabricated with a dental CAD/CAM. Ann Jpn Prosthodont Soc 2009; 1: 139-147.
14) Oshitayakul D, Lertrid W, Anatamana C, Pittayachawan P. The comparison of the marginal gaps of zirconia framework luted with different types of phosphate based-resin cements. M Dent J 2015; 35: 237-251.
15) Zortuk M, Bolpaca P, Kilic K, Ozdemir E, Agulogu S. Effect of finger pressure applied by dentists during cementation of all-ceramic crowns. Eur J Dent 2010; 4: 383-388.
16) Magne P, Razaghy M, Carvalho MA, Soares LM. Luting of inlays, onlays, and overlays with preheated restorative composite resin dose not prevent seating accuracy. Int J Esthet Dent 2018; 13: 318-332.