Original Article

Flexural strength and viscosity of dental luting composite reinforced with zirconia and alumina

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ABSTRACT

Objectives

Optimum flexural strength and viscosity are essential for longevity while efficiently handling dental luting composite. This study aims to investigate the effects of zirconia and alumina on the flexural strength (FS)and viscosity of dental cement made of silica rice husk.

Materials and Methods

Study groups with different percentages of weight (wt.): Group 1 (3 wt.% zirconia), Group 2 (3 wt.% alumina), and Group 3 (3 wt.% zirconia and 2 wt.% alumina), negative control specimen (0 wt.% zirconia/ alumina) and Rely-X U200 (3M ESPE). A universal testing machine and a rheometer were used to test FS and viscosity, respectively. One-way ANOVA and post-hoc Bonferonni test were used for data analyses.

Results and Discussion

FS and viscosity for Group 3 (3 wt.% zirconia and 2 wt.% alumina) were significantly higher compared to the negative control ($p<0.05$). ***Conclusion:*** Adding zirconia and alumina improved flexural strength without compromising the viscosity of dental luting composite. This experimental dental luting cement using hybrid fillers could be an alternative to resin luting cement.

Keywords

Dental luting composite; Alumina;Zirconia; Flexural strength; Viscosity

INTRODUCTION

Today, a tremendous demand for aesthetic and longevity restorations [1]. In addition to size, nano silica is widely used in dental composite due to the advantages of its large surface area and spherical shape [2]. Generally, the filler strengthens the matrix resin by increasing its mechanical properties [3, 4, 5]. This study used silica is obtained from biowaste of an agricultural product as the primary filler in the experimental dental luting composite. Adding other fillers may produce a more stable dental composite resin regardless of its application as direct restoration or luting cement. Zirconia and alumina are alternative fillers to combat the problem of crack propagation [6, 7, 8]. Dental fillers used in the dental composite are surface modified to improve adhesion between the inorganic filler particles and organic resin matrix using a silane coupling agent [9, 10]. Other than loading and surface modification

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of filler(s), the associated factors are the types of resin and filler that could influence polymerisation and its physicomechanical properties, as noted in the previous literature [11, 12].

Modifying dental luting cement is essential to withstand mastication forces, despite the cementation of prosthodontics material such as dental crowns, particularly in the posterior region. Adding hybrid filler or fibre could strengthen the composite matrix. A hybrid filler in dental composite produced higher flexural strength than conventional composite, according to previous studies [10, 13]. However, there is no best dental luting cement for all purposes, although the addition of hybrid filler claimed to have superior physicomechanical properties due to other factors, such as the different time intervals it is exposed to varying media of immersion, as observed in previous studies [14, 15]. For this reason, further investigation of the associated factors of dental luting cement, such as viscosity, could affect the flexural strength.

An ideal resin:filler ratio had been shown to influence polymerisation shrinkage and viscosity [16, 17]. Also, an optimum filler loading is crucial to achieving a higher flexural strength with an excellent viscosity [18]. Flexural strength is one of the important mechanical tests for dental luting composite [19, 20]. Viscosity is the relative consistency of dental luting cement that can be modified by resin and filler loading. Whereas flexural strength is measured using a three-point bend method consisting of tensile and compressive states to simulate mastication forces in the oral cavity. In contrast, viscous dental luting cement fills a gap between two surfaces to avoid microleakage that can lead to dislodgement.

An optimum flexural strength of dental luting composite is vital to withstand the forces involved in oral function [21, 22]. In addition, viscosity is another factor associated with easy mixing and polymerisation shrinkage [23]. To the best of our knowledge, there are currently no studies of flexural strength and the viscosity of dental cement using silica rice husk reinforced with zirconia and alumina in any published literature. This study aimed to investigate the effect of adding zirconia and alumina into the experimental dental luting composite. The hypotheses of this study were that adding zirconia or alumina has no effect on i) flexural strength, and ii) the viscosity of dental luting composites.

MATERIALS AND METHODS

Materials

The resins used were Bisphenol A-glycidyl methacrylate (Bis-GMA) (Esstech, Inc., Essington, PA, USA) and triethylene glycol dimethacrylate (TEGDMA) (Sigma-Aldrich, USA). The commercial products used were zirconia powder <50 nm (US Research Nanomaterials, Inc, USA) and alumina powder <50 nm (Sigma-Aldrich, USA) with silica powder extracted from rice husk. The fillers were treated with 6 wt.% γ-methacryloxypropyltrimethoxysilane (γ-MPS) (Sigma-Aldrich, USA) according to Noushad *et al.* [24]. Other materials used were DL-champhorquinone (CQ) (Merck, Schuchardt OHG, Germany), (2-dimethylaminoethyl) methacrylate (DMAEM) (Merck, Schuchardt OHG, Germany), and Rely-X U200 (3M ESPE, USA).

Preparation of experimental dental luting composite

Treated silica, zirconia, and alumina were mixed according to the formulation used for the experimental dental luting composite shown in Table 1. In this study, filler/resin (30/70) was used. The treated zirconia and alumina were added into the mixture of resins, 0.5 wt.% CQ, and 0.5 wt.% DMAEM. The filler/resin mixture was vortexed for 2 minutes and stored before testing.

Table 1 Composition of experimental dental luting composite

Groups	Filler (30 wt.%)			Resin (70 wt.%)
	Si (wt.%)	ZrO_2 (wt.%)	Al_2O_3 (wt.%)	
3 wt.% ZrO_2	97	3	-	Bis-GMA/ TEGDMA (60/40)
3 wt.% Al_2O_3	97	-	3	
3 wt.% ZrO_2 and 2 wt.% Al_2O_3	95	3	2	

Si = Silica; ZrO_2 = Zirconia; Al_2O_3 = Alumina

For comparison, 0 wt.% zirconia/alumina and Rely-X U200 (3M ESPE, USA) were used as a negative control and positive control, respectively. All specimens were subjected to a flexural strength test and viscosity test. One-way analysis of variance and Bonferroni post-hoc test were used at $p<0.05$ significance level.

Flexural strength test

A total of 40 specimens (n=8/group) were prepared for the FS test. A stainless-steel mould 25 mm in length, 2 mm in height, and 2 mm in width were used according to the International Organisation of Standardisation (ISO) 9917-2: 2017 [25, 26]. Each specimen cured three layers incrementally for 60 seconds per layer using a light curing unit (XL3000, 3M ESPE, St. Paul, MN, USA) at a wavelength of 460 mW/cm^2. All specimens were measured for an average of three values using a digital micrometer (Mitaka, Japan). The specimens were stored in distilled water overnight at 37°C before the FS test. The Instron universal testing machine (AG-X plus, Shimadzu, Japan) was used to test FS and set up at 20 kN loading force and 1 mm/min crosshead speed.

Viscosity test

A rheometer (MCR MCR 301™, Anton Paar Physica, Austria) set up at 25°C was used to test the viscosity of the dental luting composite. The rheometer was performed using a 25 mm diameter cone and plate geometry at 0.1 rad cone angle and 1 mm gap. The viscosity was evaluated using s shear sweep with different oscillation frequencies at 1 and 10 rad/s. An average of three values was recorded for each group. Then, the mean complex viscosity of the dental luting composite was obtained using an equation [27] as follows:

$$\tilde{n} = \tau / \acute{y}$$

where ñ, τ, and ý are the complex viscosity, the shear stress, and the shear strain rate, respectively.

Statistical analysis

The Statistical Package of Social Sciences (IBM, New York, United States) version 27.0 was used for data entry and statistical analysis. The level of statistical significance was set at 0.05. Data analysis was performed using a one-way analysis of variance (ANOVA), complemented by post-hoc Bonferroni significant difference multiple comparison test.

RESULTS

Flexural strength of specimens

Table 2 shows the mean values of FS with standard deviation (SD) for each group of specimens. The findings show FS was insignificant despite increased values by adding zirconia and alumina compared to the negative control (p>0.05). However, there was a significant increase with 3 wt.% zirconia and 2 wt.% alumina compared to the negative control (p<0.05). The results present a FS enhancement by zirconia and alumina, although it is lower than that of Rely-X U200.

Table 2 Flexural strength of dental luting composite

Dental luting composite	Flexural strength (MPa)	F statistic[a]	p value[a]
	Mean (SD)	(df)	
0 wt.% zirconia/ alumina 3 wt.% ZrO_2	31.34 (3.21)[c] 38.33 (6.64)[c]	23.86 (4)	< 0.001
3 wt.% Al_2O_3	34.66 (5.56)[cd]		
3 wt.% ZrO_2 and 2wt% Al_2O_3	46.15 (9.59)[bcd]		
Rely-X U200	59.17 (5.38)[b]		

Statistical analysis was carried out using One-way ANOVA[a], followed by post-hoc Bonferroni test. Significance level set at p=0.05. The same letter indicates a statistically significant difference (p<0.05). Letter b indicates statistically significant compared to 0wt% zirconia/alumina. Letter c indicates statistically significant compared to Rely-X U200. Letter d indicates a statistically significant difference.

Viscosity of specimens

Table 3 shows the complex viscosity of the study groups at different oscillation frequencies. There is a slightly different value compared to the negative control, while the negative control was significantly lower than 3 wt.% zirconia at 1 rad/s (p<0.05). However, a smaller decreased complex viscosity with 3 wt.% zirconia and 2 wt.% alumina despite not being statistically significant to the negative control at 1 rad/s. Nevertheless, the negative control was significantly higher than the 3 wt.% zirconia and 2 wt.% alumina at 10 rad/s (p<0.05).

Statistical analysis was carried out using One-way ANOVA[a], followed by post-hoc Bonferroni test. Significance level set at p=0.05. The same letter indicates a statistically significant difference (p<0.05). Letter b indicates statistically significant compared to 0wt% zirconia/alumina. Letter c indicates statistically significant compared to Rely-X U200.

DISCUSSION

Dental luting cement is used to support aesthetics and mastication [28]. For this reason, physical and mechanical properties are important for its longevity. From the findings, the FS values were 31.34 to 46.15 MPa, higher than 20 MPa, the minimum FS allowable for resin-

Table 3 Viscosity of dental luting composite with different oscillation frequency

Dental Luting Composite	Viscosity (Pa.s)	F statistic^{a}	p value^{a}
	Mean (SD)	(df)	
Complex viscosity at ω=1 rad/s			
0 wt.% zirconia/alumina	2.53 (0.73)^{c}	22.87 (4)	<0.001
3 wt.% ZrO_2	2.61 (0.64)^{c}		
3 wt.% Al_2O_3	3.46 (0.744)^{bc}		
3 wt.% ZrO_2 and 2wt% Al_2O_3	2.34 (0.21)^{c}		
Rely-X U200	7.55 (5.28)^{b}		
Complex viscosity at ω=10 rad/s			
0 wt.% zirconia/alumina	2.50 (0.90)^{c}	22.87 (4)	<0.001
3 wt.% ZrO_2	2.25 (0.31)^{c}		
3 wt.% Al_2O_3	2.73 (0.10)^{c}		
3 wt.% ZrO_2 and 2 wt.% Al_2O_3	1.89 (0.10)^{bc}		
Rely-X U200	5.41 (6.54)		

modified glass ionomer according to the ISO 9917-2: 2017 [25, 29]. In addition, the FS values of the experimental dental luting cement are comparable to PanaviaF2, a dual-cured adhesive resin cement when immersed in distilled water as in a previous study [30]. Irie *et al.* also found Panavia F produced 34.8 MPa for FS, which is close to the value obtained for 3 wt.% zirconia and 3 wt.% alumina but a lower value than the 3 wt.% zirconia and 2 wt.% alumina presented in this study [4].

Based on these findings, the type and loading of filler affect the FS values, which are in agreement with the previous studies [3, 20, 31]. Zirconia particles improved the FS of dental luting composite, which is close to the study by Beketova *et al.* in which zirconia was added in dental luting cement [5]. Whereas Souza *et al.* found alumina enhances strengthening resin matrix, which agrees with this study [7]. Adding zirconia-alumina improved the FS of dental luting composite in this study. The possible reason for this finding could be that hybrid filler sizes produce higher FS, which is close to the previous studies [31, 32]. However, an accurate polymerisation is required to achieve higher mechanical properties regardless self-adhesive cements or conventional resin cements are used [19].

A higher FS value is required for fillings compared to luting cement, according to previous studies [29, 33]. An optimum FS is important to withstand mastication forces, such as when transferring mechanical stress to the dental crown after cementation. Furthermore, a lower FS value is limited at lower mastication force [15]. For this reason, modification through the addition of filler(s) could strengthen the resin matrix to increase the longevity of restoration in the oral cavity [11, 28, 30]. Based on the data, the first null hypothesis was rejected.

Optimum mixing to ease the handling of the luting cement is another essential factor for successful dental applications [16]. Viscosity is one of the properties with which to evaluate the effect of the addition of filler(s) of different types and sizes. In this study, reinforcement with zirconia or alumina showed an increase in the complex viscosity of the study groups compared to the negative control, except for the 3 wt.% zirconia and 2 wt.% alumina group, which showed significantly lower complex viscosity than the negative control. The possible reason for this finding could be due to that a larger filler results in a void, which is close to the study by Elbishari *et al.* [34]. Hence, the second null hypothesis was also rejected. Another reason could be the resin types, where TEGDMA with or without urethane dimethacrylate (UDMA) is used to dilute Bis-GMA [35]. Different resins cause significant effect on polymerisation [36]. Nevertheless, maximum polymerisation is an essential key to increasing flexural strength without affecting by the viscosity of dental luting composite.

The results also showed a higher FS by adding 3 wt.% zirconia and 2 wt.% alumina, confirmed despite lower viscosity among the study groups. Furthermore, 3 wt% zirconia and 2 wt.% alumina produced the highest FS value. A higher degree conversion was also found for a lower viscosity of resin cement [37]. Furthermore, a sufficient degree conversion reduces polymerisation shrinkage. A mixture of different sizes of zirconia and alumina could also be associated with surface area and pore volume, which affect the degree of conversion. According to previous studies, a smaller filler size resulted in a larger specific surface area leading to void reduction [35, 38].

CONCLUSION

Within the study limitations, the conclusions include:

(1) A variety of particle filler sizes and resins improve the flexural strength and viscosity of this experimental dental luting composite.

(2) The 3 wt.% zirconia and 3 wt.% alumina experimental

dental luting composite could be an alternative to resin luting cement.

(3) An optimum zirconia-alumina filler loading and considering other resins, such as UDMA, for this experimental dental luting composite are recommended for further investigations, such as the degree of conversion and its association.

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AUTHOR CONTRIBUTIONS

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Declarations of Interest: none.

Compliance with Ethical Standards

The work is compliant with ethical standards.

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