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Influence of polymerization modes on the flexural strength and microhardness of dual cure, self adhesive resin cements: An in vitro study

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Abstract---Aim- The main aim of the study is to determine the influence of polymerization modes on the flexural strength and microhardness of three dual cure, self adhesive resin cements. Material and methods- Three self-etching/self-adhesive luting agents were mixed and dispensed according to manufacturer's instructions. The specimens for the flexural strength three-point bending test were fabricated by placing the materials into aluminum split mold that was 2 mm deep X 2 mm wide X 30 mm long. The specimens for microhardness test were made within aluminium split mold, 6 mm in diameter and 3 mm in height (6x3mm) according to ISO standards. Results- There was definite increase in flexural strength of Dual cure, self-adhesive resin cements when dual cured than self-cured. There was definite increase in microhardness of Dual cure, self-adhesive resin cements when dual cured than self-cured. Relyx U200 (group II) showed highest flexural strength when compared to BeautiCem SA (group I) and MaxcemElite (Group III). MaxcemElite (group III) showed least microhardness when compared to BeautiCem SA (group I) and RelyX U200 (group II).

Keywords---flexural strength, self adhesive resin cements, microhardness.

Introduction

Due to high esthetic appearance and high strength, esthetic resin luting agents have become popular since their introduction.¹ Dual-curing resin cements have been used for luting indirect esthetic restorations, and luting metal castings such as crowns and fixed partial dentures.² Self-adhesive resin cements which have been recently developed do not require pretreatment of the dentin, as these cements do not use an adhesive system, reduce the number of application steps, shorten clinical treatment time and decrease technique sensitivity. The dual-polymerizing resin cements combine the characteristics of both auto and light polymerization to provide cure in light attenuated areas produced by thickness and opacity of restorations and deep interproximal areas. Thus, dual cure resin cements would be the perfect choice in situations where light-cured cements are limited.⁵

flexural tests are used to evaluate the mechanical properties of dental materials. Flexural testing is an ideal method of mechanically testing brittle materials, such as composite resin luting agents. Flexural strength describes the amount of force required to bend or break the material when a test piece of specific thickness is loaded. This is called Transverse Strength.¹ Hardness is a property which is described as resistance to penetration, abrasion and scratching. It is an important parameter concerning the mastication and teeth cleaning and so it is the reason for its large application in physical characterization of dental restoration. This study was conducted to investigate the influence of different polymerization modes on flexural strength and microhardness of dual cure self-adhesive resin cements.⁴

Material and Methods

Three self-etching/self-adhesive luting agents, BeautiCemSA (Shofu), RelyX U200 (3M ESPE), Maxcem Elite (Kerr), were compared (Table 1). The materials were mixed and dispensed according to manufacturer's instructions. The specimens for the flexural strength three-point bending test were fabricated by placing the materials into aluminum split mold that was 2 mm deep X 2 mm wide X 30 mm long. For each cement, 20 specimens were fabricated. Out of these 10 were put to self-cure and remaining 10 were dual cured. The specimens for microhardness test were made within aluminium split mold, 6 mm in diameter and 3 mm in height (6x3mm) according to ISO standards. For each cement, 20 specimens were fabricated. Out of these 10 were put to self-cure and remaining 10 were dual-cured. A glass slab and a Mylar strip were placed beneath the mold. The tested luting agents were mixed according to manufacturer instructions and placed into the mold slightly overfilled. Another Mylar strip and glass slide were placed on top and pressed firmly down with finger pressure to prevent porosities from forming in the specimen. The specimens were subjected to one of the following treatments

Not Exposed to light- The specimens remained inside the dark room (x-ray room) for 40 minutes and were detached from the mold and stored in light proof containers with distilled water at 37^o C for 24 hours (Self Cured groups). Photoactivated –The specimens were light polymerized as specified by the manufacturer, with the polymerizing tip being as close as possible to the mylar surface but not touching it.The specimen were photoactivated on the upper and lower surfaces for 60 seconds, using a light cure unit (Unicorn DenMart, LED) and then detached from the mold. Following 38 minutes of final photoactivation excess material was carefully trimmed and the specimens had their surface standardized with sandpaper (P1000,WETORDRY,3M).After that the specimens were stored in light proof containers with distilled water at 37^o C for 24 hours (Dual-cured groups).²

All specimen were visually inspected. Any specimen exhibiting voids that could be reasonably assumed to adversely affect load values was eliminated from strength calculations. The Flexural strength was then tested after 24 hours using the 3-point bending method with a 25 mm span and a cross head speed of 0.5mm/ min with a universal testing machine (H50KS,Tinius Olsen).

The FS was calculated using formula:

$$\sigma = 3FL / 2bd^2$$

Where σ is the flexural strength (Mpa), F is the load at fracture (N), L is the support span (mm), b is the width (mm), and d is the depth (mm) of the specimen.³ Microhardness measurements were obtained by using Vickers Hardness Testing machine (Wilson Instruments, Model:402 MVD; Norwood). Three indentations were randomly made on the top and bottom of each specimen by applying 50 gms load for 20 seconds, and mean value for each surface was measured.

All data were analyzed statistically by two- way ANOVA test, post hoc test and student t-test.

Table 1. Materials used in the study

Resin Cement	Manufacturer	Resin Matrix Composition	Filler Loading (wt%)	Shade
RelyX U200	3M/ESPE, (STPaul, MN, USA)	Base Paste – methacrylate monomers containing phosphoric acid group, methacrylate monomer, silanated fillers, initiator components, stabilizers, rheological additives. Catalyst paste- Methacrylate monomers, alkaline fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives.	72	A2
		Paste A + Paste B-Resin matrix: GPDM,		

Maxcem Elite	Kerr (CA,USA)	comonomers, proprietary self-curing redox activator, photoinitiator CQ, stabilizer fluoroaluminosilicate glass, fumed silica, barium glass, ytterbium fluoride	67	White
BeauticemSA	Shofu (Inc, Kyoto, Japan)	Paste A : UDMA, Floroboroaluminosilicate, silicate glass, Reaction initiator and others Paste B: UDA, 2HEMA, Carboxylic acid monomer, phosphoric acid monomer, zirconium silicate, polymerization initiator and others		Ivory

Table 2. The experimental groups of this study for Flexural Strength and microhardness test

SR No	Name of Group	Mechanical Property	Activation mode	N	Total
Group 1	BeautiCem SA (shofu)	Flexural strength Test	Self Cure Dual Cure	10 10	40
		Microhardness test	Self Cure Dual Cure	10 10	
Group II	RelyX U200 (3M ESPE)	Flexural strength Test	Self cure Dual Cure	10 10	40
		Microhardness test	Self cure Dual Cure	10 10	
Group III	Maxcem Elite (Kerr)	Flexural strength Test	Self cure Dual Cure	10 10	40
		Microhardness test	Self cure Dual Cure	10 10	

Results

Table 3. Flexural Strength [Mean (MPa) and Standard Deviation]

Cement	Self cure	Dual cure
BeautiCem SA	58.51+ ₋ 6.0	74.2+ ₋ 5.9
Relyx U 200	66.7+ ₋ 5.4	81.1+ ₋ 5.5
MaxCem Elite	57.8+ ₋ 4.5	68.9+ ₋ 6.5

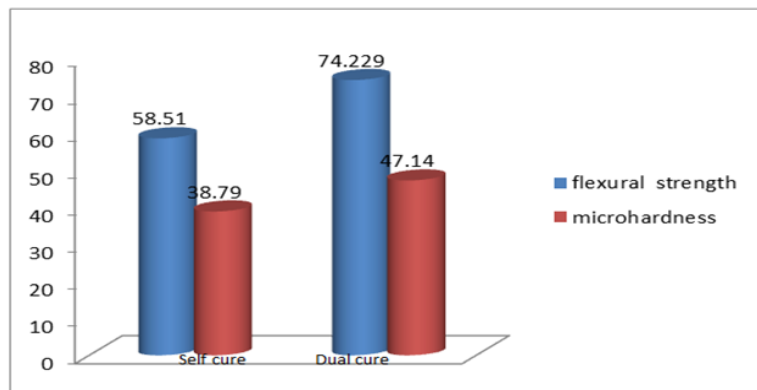
Table 4. Microhardness [Mean (MPa) and Standard Deviation]

Cement	Self cure	Dual cure
BeautiCem SA	38.7±3.7	47.1± 1.6.
Relyx U 200	37.2 ± 1.0	51.1± 3.0

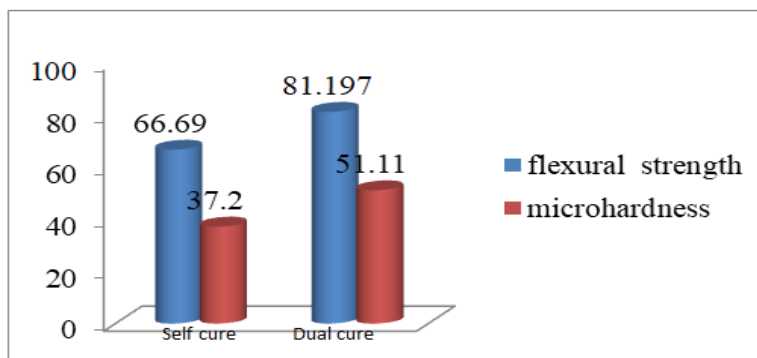
MaxCem Elite	35.5±2.7.	44.2± 3.4.
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One way ANOVA revealed highly significant difference in the mean flexural strength values and mean microhardness values of dual cured samples in all the three groups. However, there was significant difference in mean flexural strength and mean microhardness values of self cured samples in all the three groups (<0.05). Student t- test (intragroup analysis) had revealed highly significant difference in the mean flexural strength values and mean microhardness values of both self-cure and dual cure samples in Groups I, Group II and Group III (<0.05). Post- hoc bonferroni multiple comparison test revealed that Relyx U200 (group II) showed highest flexural strength when compared to BeautiCem SA (group I) and MaxcemElite (Group III). MaxcemElite (group III) showed least microhardness when compared to BeautiCem SA (group I) and RelyX U200 (group II).

Graph 1. Intragroup comparison in Group I (BeautiCem SA, shofu)



Graph 2: Intragroup comparisons in RelyX U200 (3M ESPE) (Group II)



Graph 3. Intragroup comparisons in Maxcem Elite, Kerr (Group III)

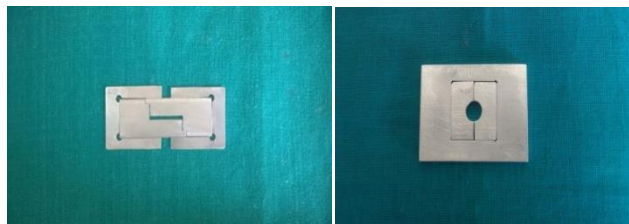
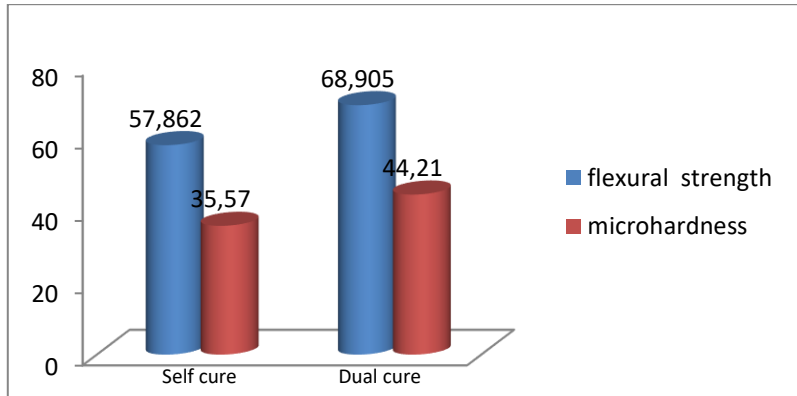


Figure 1

- a) Aluminium split mold for the preparation of Flexural strength test specimens
- b) Aluminium split mold for the preparation of Vickers microhardness test specimens



Figure 2 a) Group I. Flexural strength specimen (self cure) (b) Flexural strength specimen (dual cure) (c) Microhardness specimen (self cure) (d) Microhardness specimen (dual cure)



Figure 3 a) Group II. Flexural strength specimen (self cure) (b) Flexural strength specimen (dual cure) (c) Microhardness specimen (self cure) (d) Microhardness specimen (dual cure)

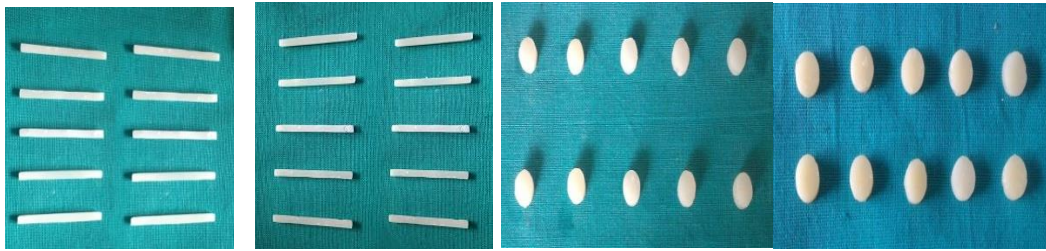


Figure 4 (a) Group III. Flexural strength specimen (self cure) (b) Flexural strength specimen (dual cure) (c) Microhardness specimen (self cure) (d) Microhardness specimen (dual cure)

Discussion

This study showed that dual curing polymerization methods produce higher flexural strength than self-curing method on dual-cure resin cements. There was a significant variation in the results of the three-point-bending tests of the 3 dual cure resin materials. All the tested self-adhesive dual cure resin cements had greater flexural strength when dual cured than self-cured specimens. The RelyX U200 (Group II) showed highest mean flexural strength of 66.69 Mpa when self-cured and 81.98 Mpa when dual cured among all the groups. The Maxcem Elite (group III) showed least mean flexural strength of 57.86 Mpa and 68.90 Mpa when self-cured and dual cured respectively. In case of BeautiCem SA (Group I) the average flexural strength for self-cured and dual cured subgroups were 58.51 Mpa and 74.22 Mpa respectively. Based on the results it was found that all dual curing resin cement, when chemically activated (self-cured) showed lower flexural strength values than photoactivated (dual cured).

These results were consistent with other studies. Guimaraes et al. showed that light curing polymerization methods produce higher flexural strength and modulus than self-curing method on dual cure resin cements. The resin cements Relyx ARC and Relyx U100 showed 48% reduction in values of flexural strength and modulus of elasticity when left to be self-cured. Clearfil Esthetic cement and Clearfil SA cement showed reduction of around 42-42% (respectively) in the same parameters. Probably the reduction of mechanical properties happens because the initiation of chemical reaction (induced by the production of free radicals) is delayed by the manufacturers in order to let enough time for manipulation until it achieves its end. This is possible by the addition of large amounts of inhibitors on resin cements composition. Thus, this manufacturer's intervention possibly led to decrease mean values of mechanical properties of the resin cements when left to be self-cured.⁹ Attar et al. studied mechanical and physical properties of five contemporary dental luting agents and found that photopolymerization of resin based cements was necessary to maximize strength and rigidity; therefore areas where light cannot reach all of the cement may have reduced mechanical properties.⁷ Under micro-hardness, there was a significant variation in the results of microhardness test tests of 3 dual cure resin materials. All the tested self-adhesive dual cure resin cements had greater microhardness when dual cured than self-cure specimens. The Relyx U200 (Group II) showed highest mean microhardness of 51.11 HV when dual cured among all the groups. In case of

BeautiCem SA (group 1) the average microhardness for self-cured and dual cured subgroups were 38.79 HV and 47.14 HV respectively. Similarly for Maxcem Elite (group III) the average microhardness value for self-cured and dual cured groups were 35.57 HV and 44.21 HV respectively. El- Mowafy and Ruboand Hofmann et al found that the self-curing components of most dual- cured resin cements were not capable of compensating the light attenuation and reported reduction in hardness and flexural strength values. Peutzfeldt, Rueggeberg and Caughman and Mowafy et al verified that when dual cured cement had been light- cured, there was an increase of conversion degree compared with dual – cured cement polymerized only by chemical activation. The difference in microhardness values among products in self-curing mode cannot be attributed solely to difference in monomer conversion, as differences in monomer composition and content of filler particles play an important role in the mechanical properties of resin- based materials.⁶ There were several limitations in the simulation of clinical situations in this study. They pertained to irradiation duration, light intensity, light transmission, property of restorative materials, restoration thickness and shade. These factors influenced the polymerization efficiency of dual cure resin cements, but which were not adequately simulated and evaluated in this study. Therefore, further studies – which better simulate these clinical situations, are necessary for a thorough evaluation of the polymerization degree of dual cure resin cements placed under restorations.⁸

Conclusion

Respecting the limitations of our study, the following conclusions are drawn-

1. There was definite increase in flexural strength of Dual cure, self-adhesive resin cements when dual cured than self-cured.
2. There was definite increase in microhardness of Dual cure, self-adhesive resin cements when dual cured than self-cured.
3. Relyx U200 (group II) showed highest flexural strength when compared to BeautiCem SA (group I) and MaxcemElite (Group III). MaxcemElite (group III) showed least microhardness when compared to BeautiCem SA (group I) and RelyX U200 (group II).

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