

## RESEARCH ARTICLE

# Evaluation of Temporization Period with Zinc Oxide Eugenol and Non-eugenol Cement on Bond Strength of Self-adhesive Dual-cure Resin Cement

<sup>1</sup>Paul Pudukadan, <sup>2</sup>Julie G Alapatt, <sup>3</sup>Noxy G Manjuran, <sup>4</sup>Jaini Lalithamma, <sup>5</sup>Vidya K Shenoy, <sup>6</sup>Edwin Kunnamkumarath

## ABSTRACT

**Context:** Eugenol-based cements are used for temporization; however, they affect the physical properties of resin cements.

**Aim:** To evaluate effect of eugenol and non-eugenol-containing temporary cements on the tensile bond strength of self-adhesive dual-cure resin cement (SARC) at intervals of 24 hours, 7 days, and 14 days.

**Settings and design:** Preclinical *in vitro* material study.

**Materials and methods:** A total of 105 freshly extracted, intact, maxillary premolars were divided into seven groups consisting of 15 teeth each followed by preparation to receive cast metal crowns. They were temporized using eugenol cement in groups II, III, and IV, non-eugenol cement in groups V, VI, and VII, and stored in distilled water for specific periods. Provisional cement was removed using ultrasonic scaler and cleaned with pumice-water slurry. Thereafter, the cast crowns were cemented using SARC.<sup>46</sup> The tensile bond strength of SARC in the respective groups were tested on the universal testing machine using "Crown pull test" at a cross-head speed of 0.5 mm/min until failure.

**Results:** Results were analyzed using analysis of variance (ANOVA) and Tukey's *post hoc* test. After 24 hours, significant reduction in the bond strength was observed in both eugenol and non-eugenol groups. After 7 and 14 days, the reduction in the bond strength was insignificant. No significant differences were found in the bond strengths of SARC among eugenol and non-eugenol groups.

**Conclusion:** The study showed that waiting for a week before performing cementation with SARC when using eugenol or non-eugenol provisional cements displayed favorable results.

**Keywords:** Non-eugenol cement, Provisional restoration, Self-adhesive dual-cure resin cement, Temporization, Zinc oxide eugenol cement.

**How to cite this article:** Pudukadan P, Alapatt JG, Manjuran NG, Lalithamma J, Shenoy VK, Kunnamkumarath E. Evaluation of Temporization Period with Zinc Oxide Eugenol and Non-eugenol Cement on Bond Strength of Self-adhesive Dual-cure Resin Cement. *Int J Oral Care Res* 2018;6(1):69-76.

**Source of support:** Nil

**Conflict of interest:** None

## INTRODUCTION

An ever-increasing demand for cosmetic restorations and advances in adhesive dentistry has led to an increase in the choice of resin luting cements for indirect restorations. These cements bond to the tooth structure and restorations have higher compressive, flexural, and tensile strengths than conventional cements.

Newer self-adhesive resin cements have their monomers and adhesives incorporated in the cement itself, thereby eliminating the need for pretreatment procedures, provided the bonding substrates are clean and free from fluid contamination. These cements contain both a self-cure initiator (benzoyl peroxide) and a light-cure initiator (camphoroquinone). Although chemical reaction alone ensures curing, light curing enables a higher degree of polymerization.<sup>1</sup>

Indirect restorations require temporization to provide optimal esthetics, sound articulation in the patient's mouth, maintain periodontal health, prevent displacement of teeth, and ensure pulp protection. Certain clinical situations demand long-term provisional restorations where a luting agent with good mechanical properties, low solubility, and good adhesion is critical to avoid problems with cement washout, marginal leakage, bacterial infiltration, and caries.

Provisional cements routinely used are: zinc oxide eugenol (ZOE) and zinc oxide non-eugenol (ZONE) cements. Eugenol penetrates and diffuses through the dentin, affecting the bond strength of resin luting agents. Hence, its bonding efficiency on both the dentin substrate and definitive restorations are compromised. It has excellent antibacterial effects also.<sup>2</sup> The ZONE provisional luting cements replace eugenol with various types of carboxylic acids. They have greater retention compared with ZOE cements, but no sedative effect on the pulp.<sup>3</sup>

<sup>1</sup>Senior Lecturer, <sup>2</sup>Associate Professor, <sup>3,4</sup>Reader, <sup>5</sup>Professor and Head, <sup>6</sup>Consultant Prosthodontist

<sup>1,3</sup>Department of Prosthodontics, P.S.M. College of Dental Science & Research, Thrissur, Kerala, India

<sup>2</sup>Department of Prosthodontics, Government Dental College Thrissur, Kerala, India

<sup>4</sup>Department of Prosthodontics, Amrita School of Dentistry Amrita University, Kochi, Kerala, India

<sup>5</sup>Department of Prosthodontics, A.J. Institute of Dental Sciences Mangaluru, Karnataka, India

<sup>6</sup>Trinity Dental Clinic, Thrissur, Kerala, India

**Corresponding Author:** Julie G Alapatt, Associate Professor Department of Prosthodontics, Government Dental College Thrissur, Kerala, India, e-mail: drjialapatt@gmail.com

Studies show that residues of ZOE and ZONE provisional cements have shown to reduce the tensile bond strength of resin luting agents. Routine procedures before definitive cementation, such as mechanical cleaning using pumice, water spray, and acid conditioning of dentin, would neutralize the inhibitory effect of provisional cement on bond strength. However, contradictory literature reports exist on the influence of ZOE on bond strength of adhesive systems to dentin.<sup>4</sup>

Another contributing factor can be the variable time interval that the ZOE cement remains in contact with the dentin surface prior to definitive cementation. In several studies, the exposure time to ZOE has ranged from 24 hours to 10 days or 4 weeks, making it difficult to comprehend the studies and determine the actual effect of eugenol on bond strength of dental adhesives.<sup>4</sup>

Furthermore, the microshear or microtensile bond strength values have been evaluated on relatively small surface areas.<sup>5</sup> Therefore, the current study was undertaken to evaluate and compare the effect of macro-tensile bond strength of SARC, on exposure to ZOE and ZONE cements, at different time intervals. The null hypothesis to be tested was that exposure time of ZOE will not affect the bond strength value (Flow Chart 1).

## MATERIALS AND METHODS

A total of 105 freshly extracted human maxillary premolars were collected, thoroughly cleansed under tap

water, and stored in distilled water. Healthy premolars with intact clinical crowns, and sufficiently wide occlusal surfaces were included. Decayed, restored, and deciduous teeth, as well as teeth with deep cervical abrasions and arrested caries, developmental anomalies, heavy attrition, were excluded. All the samples were prepared by the same operator, under standardized conditions, to minimize subjective errors.

A hole was drilled in the center of radicular 2/3rd of each tooth to strengthen the bond with the acrylic, while mounting the specimens in self-cure acrylic resin blocks. Surveyor was employed to centralize the long axes of each tooth sample perpendicular to the horizontal plane, and orient cemento-enamel junction 1 to 2 mm coronal to the acrylic resin block of 2 × 2 × 5 cm. The samples were mounted on the surveying table at 0° tilt. Tooth preparation was done using a straight micromotor hand-piece (NSK, Japan), and tapering round diamond point (TR 12, MANI, Inc) to uniformly reduce 0.5 to 1 mm of the tooth structure with standard 6° taper and a chamfer finish line under constant irrigation (Fig. 1).

An irreversible hydrocolloid impression (Algitex, Alginate DPI Ltd, India) was made, poured in dental stone (Next, Next Dental Products, India), and provisional crowns were fabricated using tooth-colored autopolymerizing acrylic resin (DPI Ltd, India). Autopolymerizing custom acrylic resin trays were used to make definitive polyvinyl siloxane impression (Speedex, Coltene, and Whaledent, UK) using putty/light-body dual viscosity technique (Fig. 2). Wax patterns were made on dies (Denstone Plus, Pankaj Ltd, India) with flat occlusal anatomy and sprued with 2.5 mm diameter wax (Renfert, Germany). This sprue extension was axially oriented, facilitating mounting on the universal testing machine (The Shimadzu Autograph AGS-X series, Japan). Induction casting technique (Fornax, Bego, Germany) was used to fabricate definitive metal crowns (Girobond,

Flow Chart 1: Investigation design

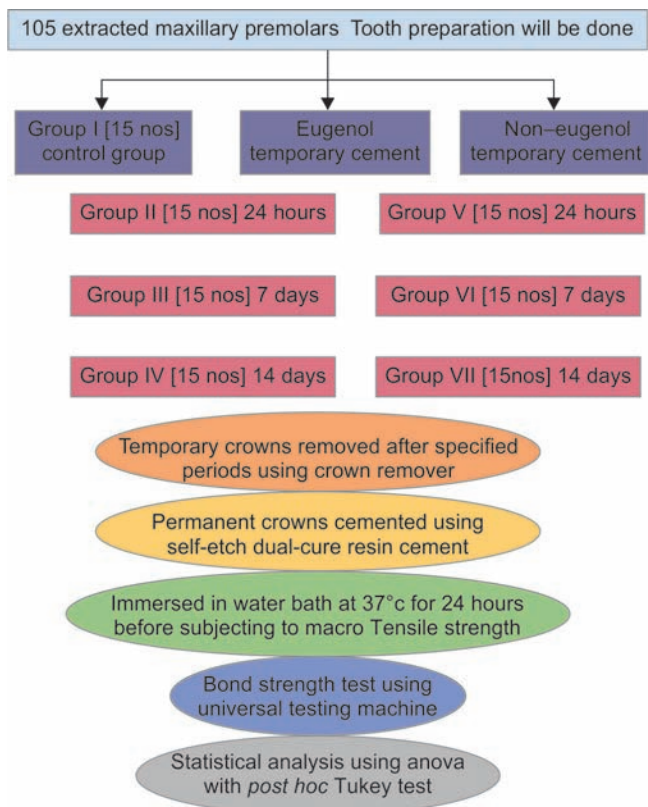
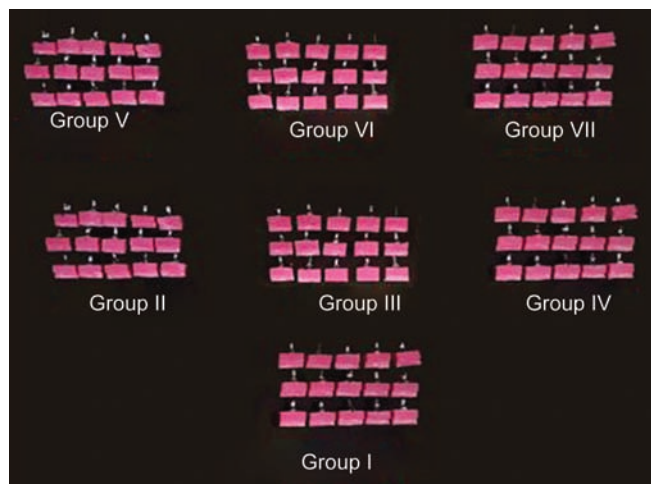


Fig. 1: A total of 105 samples, seven groups of 15 each



**Fig. 2:** Tooth preparation using dental surveyor



**Fig. 3:** Completed casting with sprue extensions



**Fig. 4:** Removal of provisional cement using scaler



**Fig. 5:** Removal of cement remnants using pumice slurry

Amanngirbach, Germany) and sandblasted with 50  $\mu$  alumina, finished, and polished (Fig. 3).

The samples were then divided into seven groups of 15 teeth each. In group I (control group), the definitive crowns were directly luted with SARC, without any temporization. All other study groups were temporized with either ZOE or ZONE as mentioned in the study design chart. A mixing pad and stainless steel spatula were used for mixing both eugenol and non-eugenol zinc oxide-based cements, to obtain a creamy consistency for the provisional crown cementation. After this, they were kept in a water bath at 37°C for the specified periods (Figs 4 and 5).

Once the provisional crowns were removed, the residual provisional cement was cleared away with an ultrasonic scaler (UDS-J, Woodpecker, China) followed by cleaning with pumice-water slurry (White Gold, Asia dental; India) using a slow speed hand-piece (NSK, Japan), rinsed with air-water stream and dried (Figs 6 to 8).

Definitive luting of metal crowns was done using SARC (Rely X U200, 3M, Germany) by dispensing the catalyst and base paste simultaneously in 1:1 ratio from the



**Fig. 6:** Permanent cementation using SARC

clicker and mixed on a mixing pad using agate spatula.<sup>6</sup> After luting of definitive crowns, all the specimens were kept in 37°C water bath for 24 hours before subjecting them to the macrotensile test. The acrylic base of the samples was secured to the lower clamp and the straight



Fig. 7: Samples mounted before testing



Fig. 8: Samples after testing

cast metal sprue extension on the occlusal surfaces to the upper clamp of the universal testing machine. The cross-head speed was set at 0.5 mm/min until failure. After this, the bond strengths of SARC in the respective groups were tested using “Crown pull test” by moving the upper member away from the lower member.

**RESULTS**

The bond strength values obtained after testing the specimens were statistically analyzed using analysis of

variance (ANOVA), with *post hoc* test using Statistical Package for the Social Sciences software version 20.0 (SPSS Inc., Chicago, IL, USA). The data were expressed as maximum tensile stress in megapascals. All statistical tools gave a significant ( $p < 0.05$ ) value (Tables 1, 2 and Graph 1).

After applying ANOVA and *post hoc* Tukey test between the groups, it was seen that both the tools showed a statistically significant reduction in macrotensile bond strengths among the eugenol and non-eugenol

**Table 1:** Mean macrotensile bond strength

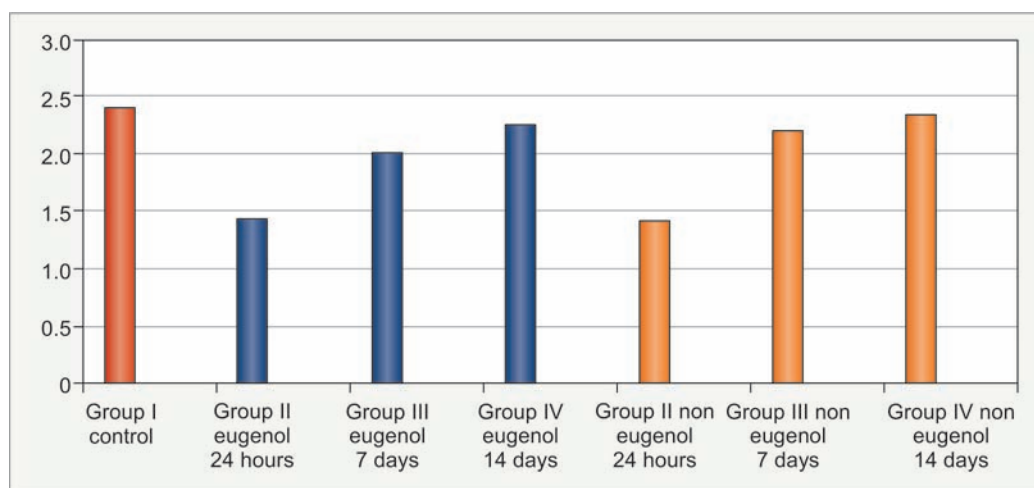
Resin cements n = 15 in each group	Mean (MPa)	Std. deviation	Min (MPa)	Max (MPa)	Confidence interval	
					Lower 95% CI	Upper 95% CI
Control group I	2.401	0.7757	1.101	3.735	1.847	2.955
Eugenol 24 hours group II	1.430	0.2735	0.272	2.344	1.235	1.625
Eugenol 7 days group III	1.986	0.7818	0.6912	3.008	1.534	2.545
Eugenol 14 days group IV	2.259	0.7743	0.7677	3.295	1.706	2.812
Non-eugenol 24 hours group V	1.412	0.6171	0.1462	2.144	0.971	1.853
Non-eugenol 7 days group VI	2.204	0.85	0.4394	3.049	1.597	2.811
Non-eugenol 14 days group VII	2.481	0.3685	1.614	2.834	2.218	2.744

CI: Confidence interval

**Table 2:** Comparison of bond strengths at different exposure times

Interval	Groups	n	Mean	Std. deviation	f-value	p-value
24 hours	Control	15	2.401479	0.775739	14.959	0.001*
	Eugenol	15	1.43095	0.273458		
	Non-eugenol	15	1.411693	0.617148		
	Total	45	1.748041	0.741001		
7 days	Control	15	2.401479	0.775739	0.669	0.52
	Eugenol	15	1.985988	0.781843		
	Non-eugenol	15	2.204033	0.84998		
	Total	45	2.197167	0.794019		
14 days	Control	15	2.401479	0.775739	0.286	0.754
	Eugenol	15	2.2591	0.774272		
	Non-eugenol	15	2.481849	0.368505		
	Total	45	2.380809	0.650943		

\*Statistically significant ( $p < 0.05$ ), ANOVA applied. After applying ANOVA test between the groups at different intervals, it was seen that there was a statistically significant reduction in bond strengths among the eugenol and non-eugenol groups against the control, observed at 24 hours ( $p$ -value = 0.001). At 7 and 14 days the reduction in bond strength observed was statistically insignificant



**Graph 1:** Mean bond strengths of the eugenol group and non-eugenol group after intervals of 24 hours, 7 days, and 14 days against the control group

groups against the control, observed at 24 hours (p-value = 0.001; 0.003 respectively), whereas at 7 and 14 days, the reduction in bond strength observed was statistically insignificant (Tables 3 and 4).

The three eugenol groups and the three non-eugenol groups were paired among themselves, and paired t-test was used to compare the macro-tensile bond strength values between the groups. It was seen that there was

**Table 3:** Intergroup comparison using *post hoc* Tukey test

Dependent variable	(I) group	(J) group	Mean difference (I-J)	Std. error	p-value
24 hours	Control	Eugenol	0.970*	0.265509	0.003*
		Non-eugenol	0.989*	0.265509	0.003*
	Eugenol	Non-eugenol	0.019	0.265509	0.997
7 days	Control	Eugenol	0.415	0.359214	0.489
		Non-eugenol	0.197	0.359214	0.848
	Eugenol	Non-eugenol	0.2180	0.359214	0.818
14 days	Control	Eugenol	0.1423	0.298559	0.883
		Non-eugenol	0.0803	0.298559	0.961
	Eugenol	Non-eugenol	0.2225	0.298559	0.739

\*Statistically significant ( $p < 0.05$ ), *post hoc* Tukey test applied. After applying *post hoc* Tukey test between the groups at different intervals, it was seen that there was a statistically significant difference in bond strengths among the eugenol and non-eugenol groups against the control, observed at 24 hours (p-value = 0.003)

**Table 4:** Intragroup comparison using paired t-test

Group	Pair	Duration	Mean	n	Std. deviation	Paired differences		t-value	p-value
						Mean difference	Std. deviation		
Eugenol	Pair 1	24 hours	1.43095	15	0.273458	0.55504	0.761214	2.306	0.047*
		7 days	1.985988	15	0.781843				
	Pair 2	24 hours	1.43095	15	0.273458	0.82815	0.829323	3.158	0.012*
		14 days	2.2591	15	0.774272				
	Pair 3	7 days	1.985988	15	0.781843	0.27311	0.630475	1.37	0.204
		14 days	2.2591	15	0.774272				
Non-eugenol	Pair 1	24 hours	1.411693	15	0.617148	0.79234	1.047108	2.393	0.04*
		7 days	2.204033	15	0.84998				
	Pair 2	24 hours	1.411693	15	0.617148	1.07016	0.685774	4.935	0.001*
		14 days	2.481849	15	0.368505				
	Pair 3	7 days	2.204033	15	0.84998	0.27782	0.536181	1.638	0.136
		14 days	2.481849	15	0.368505				

\*Statistically significant ( $p < 0.05$ ), paired t-test applied. After applying paired t-test between the groups at different intervals, it was seen that there was a statistically significant difference in bond strengths among the eugenol Pair 1 (p-value: 0.047) and Pair 2 (p-value: 0.012) and non-eugenol Pair 1 (p-value: 0.04) and Pair 2 (p-value: 0.001) groups

a statistically significant difference in bond strengths among the eugenol pair 1 (p-value: 0.047) and pair 2 (p-value: 0.012) and non-eugenol pair 1 (p-value: 0.04) and pair 2 (p-value: 0.001) groups.

## DISCUSSION

The number of choices for indirect restorations has evolved greatly over the last decade. The long-term success, including the retention and marginal integrity of a restoration, is heavily dependent on the proper selection and manipulation of dental cements.<sup>7,8</sup> Resin cements are commonly employed in cases with concerns of retention and esthetics due to their high compressive and tensile bond strengths, low solubility, and esthetics. No currently available dental cement is ideal for all situations.<sup>1</sup>

Resin cements can be divided into three subtypes based on bonding mechanism (total-etch, self-etch, and self-adhesive).<sup>7</sup> The SARC does not require any surface pretreatment or bonding agents to maximize their performance, whereas the other two categories require multiple technique-sensitive steps. Their primers contain acidic polymerizable monomers which dissolve or incorporate the smear layer into the bonding interface,<sup>9</sup> and bonding to the dentin/enamel surfaces is based on micromechanical interlocking of the adhesive resin.<sup>10</sup> They also contain fillers and initiator systems.<sup>4,11,12</sup> Self-adhesive cement, Rely X U200, a dual-cure two-paste system was used in the present study.

Several factors might interfere with the bonding ability of adhesive systems to enamel or dentin, including the adhesion strategy, conditioning time, solvent removal method, thickness of the adhesive layer, substrate structure, and even the provisional restorative material previously used.<sup>2,13-16</sup>

Most of the prosthodontic restorations require a provisionalization phase after tooth preparation which protects the dentin-pulp complex from physical-chemical stimulus prior to definitive restoration.<sup>2</sup> Among them, ZOE is widely used. The end product of the setting reaction between zinc oxide and eugenol produces zinc eugenolate, which is unstable in the presence of water, readily undergoes hydrolysis with the release of free eugenol that is initially rapid, and then decreases exponentially, as all the surface eugenol is hydrolyzed. Eugenol being a phenolic compound is insoluble in water and because of its formulation, it inhibits resin polymerization which alters many physical properties of resin including adverse effects on surface roughness, transverse strength, and surface hardness.<sup>17</sup> According to Farah and Powers,<sup>18</sup> an ideal provisional cement should exhibit the following characteristics: easy removal of excess cement from around the margins; good marginal seal to help minimize sensitivity; good retention, but easy removal of the

temporary prosthesis; low solubility in oral fluids; and compatibility with provisional resin restorations, resin core materials, bonding agents, and permanent cements.<sup>19</sup>

Resin polymerization can be inhibited by any material that reacts with free radicals. Eugenol is a free radical scavenger, inhibiting polymerization either by a decrease in the rate of initiation or an increase in the rate of termination,<sup>17</sup> which leads to increased surface roughness, reduced microhardness, and color stability of resin composites cured in contact with ZOE cement.<sup>11</sup>

Nasreen et al<sup>20</sup> in their study reported that eugenol causes release of calcium from dentin due to its complexing properties. This may have a softening effect on dentin. Inadequate polymerization coupled with softening of dentin leads to decreased bond strength and increased microleakage, resulting in clinical complications, such as fractured restoration, hypersensitivity, secondary caries, and surface discoloration.

The American Dental Association (ADA) specification No 30 for ZOE restorative materials lists four types. Type I cements used for provisional cementation<sup>12</sup> release more eugenol, which can be responsible for the lower resin-dentin bond strengths. The wetter ZOE mix has significantly higher diffusion rates<sup>21,22</sup> and may be more susceptible to hydrolysis. Since water cannot penetrate the set bulk materials, only dentinal tubule fluid has an effect on the rate of release of eugenol toward the pulp.

Mechanical removal of provisional cements is not 100% effective; even microscopic remnants of ZOE may<sup>4,12</sup> change the wettability, permeability, and reactivity of dentin, and alter the contact angle of liquids, such as adhesives.<sup>9</sup> The routine procedures required for adhesive cementation, such as mechanical cleaning, pumice, water spray, and acid conditioning of dentin, would neutralize the inhibitory effect on bond strength by reducing the amount of residual particles of provisional cement.<sup>9</sup> Grasso et al<sup>23</sup> showed that pumice cleansing was more effective than other cleansing techniques, such as explorer/air-water technique or with 0.12% chlorhexidine gluconate.<sup>21</sup>

In the current study, combination of these approaches has been proven effective in removing the residual provisional material and permitting bond strength similar to that achieved without the use of provisional restorations.<sup>21</sup> The ZOE and ZONE provisional luting agents were used for a period of 24 hours, 7 days, and 14 days, as the amount of residual eugenol left behind may vary at a given point of time and may affect the bond strength.

The samples were tested for macrotensile bond strength using universal testing machine since the bonding area is larger than 3 mm.<sup>5,9</sup> Darr and Jacobsen<sup>24</sup> have stated that dual curing agents require 24 hours to reach maximum cure. There was rapid increase in the hardness

followed by a steady increase over the next 24 hours. The nonirradiated, chemically cured samples exhibited steadily increasing hardness over 24 hours, but were too soft to test in the initial 30 minutes. Hence, test was performed 24 hours following permanent cementation.

According to Klocke and Kahl-Nieke,<sup>25,26</sup> the cross-head speed variation between 0.1 and 0.5 mm/min does not seem to influence debonding force measurements or failure mode of orthodontic brackets bonded to enamel with a composite adhesive. Hence, cross-head speed of 0.5 mm/min was used in the current study.

In the present study, highest mean bond strength (2.40 MPa) was obtained within the control group devoid of provisional cementation. Among II, III, and IV groups, as well as the V, VI, and VII groups, least bond strength values were obtained in the 24-hour group (1.43, 1.41 MPa) followed by a gradual increase in bond strength after 7 days (1.98, 2.20 MPa) and 14 days (2.26, 2.48 MPa) respectively. Thus, the null hypothesis that the exposure time of ZOE does not affect the bond strength of SARC was rejected. The highest bond strength values observed in group I may be due to unimpaired adhesion of SARC, resulting in the infiltration of resin into the dentin and form a true hybrid layer by simultaneous etching. Similar results were observed in related studies.<sup>9,13,22</sup> Hume<sup>3</sup> found the concentration of eugenol in aqueous phase to be in the order of  $10^{-2}$  M just beneath the ZOE cement, and  $10^{-4}$  M adjacent to the pulp.<sup>21</sup> The eugenol in the residual cement could penetrate dentin to change its wettability and reactivity.<sup>12</sup>

Initial reduction of bond strength of resin cements with the usage of eugenol-free cements was attributed to the presence of residual cement.<sup>16,27</sup> Statistically significant reduction in the bond strength values ( $p = 0.001$ ) after an exposure time of 24 hours in eugenol as well as non-eugenol groups suggests the influence of diffusion of eugenol, and the influence of oils and glycerin on the resin polymerization. It has been shown that diffusion rates of eugenol released from ZOE increased to a peak after 24 hours (about 0.3 nmol/min) and then decreased slowly to 0.08 nmol/min after 14 days, thereby improving the bond strength.<sup>13</sup> Therefore, it is expected that eugenol concentration in dentin from 7 to 14 days will not significantly affect the bond strength compared with 24 hours, as eugenol concentration is reduced to non-inhibitory levels. This explains the significant increase in bond strength at 7 and 14 days. There was no significant difference in the bond strength of SARC among ZOE and ZONE. Therefore, it seems more logical to use eugenol and non-eugenol containing provisional restoration for a week or longer.

Studies with conflicting results on microtensile bond strengths and macrotensile bond strengths exist in the

literature.<sup>9,13</sup> Apart from the choice of restorative materials, clinical outcomes may be influenced by factors, such as tooth preparation, preparation coarseness, type of luting agent, fit of restoration, type of provisional cement, duration of temporization, and also techniques used to remove the remnants of provisional cements.<sup>9,20,21</sup>

*In vitro* studies are not an accurate representation of clinical situations with regard to the amount and nature of load applied. Failure of intermediate restorations may be due to not only monoaxial loading (such as repetitive loading) but also thermal changes occurring in the mouth, both of which have not been simulated in this study. Also, larger sample sizes and additional studies are necessary to evaluate other exposure times.

There is scope for further studies using different pretreatment protocols and by using criteria that best simulate the oral environment. Other parameters, such as transverse bend strength, surface hardness, and surface discoloration and roughness can be tested for more comprehensive results.

## CONCLUSION

The bond strength of SARC significantly reduced after an exposure time of 24 hours in case of both eugenol- and non-eugenol-based provisional cements, whereas after 7 and 14 days, there was a gradual increase. Within the limitations of this study, it was concluded that there was no significant difference in the bond strength of SARC among eugenol- and non-eugenol-based provisional cement groups. It is, however, more prudent to wait for 7 days before performing definitive cementation with SARC after the usage of eugenol- or non-eugenol-based provisional cements for interim restorations.

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