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Research Article

Characterization of Polylysine Enriched Self-Adhesive Resin Cements

Zainab M. Mansi[1] **and Abdulla MW Al-Shamma**[2]

[1]*Department of Dentistry, Dijlah University College, Baghdad, Iraq*
[2]*Restorative and Aesthetic Dentistry Department, College of Dentistry, University of Baghdad, Baghdad, Iraq*

Correspondence should be addressed to Zainab M. Mansi; zainab.mah@duc.edu.iq

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Aim. The aim of this study is to add polylysine (PLS) particles to two different types of self-adhesive resin cement (RelyXU200 and Breeze) and to characterize them before and after PLS incorporation. *Materials and Methods*. Four PLS concentrations (0.2%, 1%, 2%, and 5%) were selected for incorporation into two self-adhesive resin cements (RelyXU200 and Breeze), thus the groups of this study include one control group and four experimental groups for each cement type. Different characterization tests were performed including Fourier transform infrared spectroscopy (FTIR), degree of conversion (DC), compressive strength (CS), and ISO tests 4049-2019 that include: film thickness, setting time, water sorption (WS), and solubility (SL). The statistical procedure used includes analysis of variance test (ANOVA) and multiple pairwise comparisons Tukey post hoc test which was used for multifactorial analysis. *Results*. FTIR showed that PLS addition did not change the functional groups' peaks for either cement type indicating that no chemical reaction with and/or alteration within the cement has occurred. In general, PLS addition increased the water sorption, solubility, and film thickness, it also increased the setting time for Breeze while decreasing it for RelyXU200 and all these parameters were within 4049-2019 ISO Specification. Statistically, PLS addition did not significantly change these properties compared to the control groups except for 5% PLS. Similarly, the degree of conversion and compressive strength was slightly reduced with no significant difference to the control cement groups except for 5% PLS concentration. *Conclusions*. Newly developed PLS incorporated self-adhesive resin cement exhibited remarkable mechanical and physical properties compared to control self-adhesive resin cement and passed the ISO standardization. PLS-incorporated resin cement with less than 5% have no negative impact on the physical and mechanical properties of the studied cement.

1. Introduction

Adhesive materials have dramatically transformed dentistry by conserving tooth structure due to the less invasive procedures and allowing bonding between indirect restoration and the tooth. The introduction of self-adhesive resin cement (SARCs) was promoted as having a shorter application time and a lower method sensitivity. SARCs infiltrate tooth tissues and react chemically with hydroxyapatite crystals and phosphate methacrylates. According to manufacturers, self-adhesive agents contain innovative polymerization technology in an acid environment. It is recommended for full or partial indirect restorations ceramics and composites. Self-adhesive cement can alter and integrate the smear layer into the hybrid layer. This is attributable to one of its constituents, the multifunctional acid methacrylate (carboxylic or phosphoric) [1].

Due to its acidity, it can demineralize dental substrate and facilitate the entrance of resinous cement components into the dentin matrix. The initial action of corrosive monomers on enamel and dentin promotes demineralization and infiltration of the cementing agent, resulting in a micromechanical retention field [2]. Secondary reactions between self-adhesive cement and hydroxyapatite are also essential through chemical bonds with calcium ions [3]. The best dental cement for luting purposes should be biocompatible, prevent cavities or plaque development, has low solubility, correct film thickness, extended working time, and quick setting time. Additionally, it needs to be firm.

Secondary dental caries is the leading cause of indirect restoration failure, making it risky. Plaque and biofilm of various microorganisms may easily form secondary caries in restoration margins or gaps. Therefore, luting cement with antibacterial properties is a good choice to prevent secondary decay at the tooth restoration interface [4]. Polylysine (PLS) is a cationic, naturally occurring polypeptide produced as an extracellular material by *Streptomyces albulus*. Shime and Sakai [5] made the first official identification of it in 1970. It is manufactured industrially by fermentation mutant strains of *Streptomyces albulus* as a food preservative. The natural polypeptide PLS, which is composed of L-lysine units ($n = 25–30$) [6], is degradable, water-soluble, nontoxic, and palatable and is generally recognized as safe (GRAS). PLS antimicrobial properties are well-known in food industries and are more frequently used in the biomedicine [7].

It is noteworthy that few previous studies have utilized PLS for dental application purposes. In one study, PLS incorporation in smart composites with monocalcium phosphate increased bond strength with self-adhering properties to dentin [8]. Also, Guo et al. [9] used PLS with sodium alginate coating loading nanosilver to improve the antibacterial effect and induce mineralization of dental implants.

Hence, for clinical long-term success, it is important that the cement material exhibits mechanical stability, since the restoration daily subjected to mechanical forces such as mastication, therefore, in order to assess the ability of the material to withstand these types of stress, mechanical tests such as compressive strength is important to allow complete seating of the prosthetic restorations, it must obtain an appropriate flow rate maintaining a minimum film thickness, reduced cement film thickness can also decrease the marginal discrepancies, which in turn reduce the plaque accumulation, periodontal disease, and cement dissolution [10]. Failures of cemented restorations may be explained by water sorption, solubility, and microleakage of resin cements [11]. Therefore, it is important to understand the mechanisms of water sorption and solubility of resin cements [12]. When water degrades the filler—matrix interface it acts as a plasticizer of the cement and this might lead to swelling and decreased mechanical properties [11]. However, the setting mechanism of the cements may be influenced by changes in the chemistry of their component [13]. The implications of such changes on the mechanical properties of the resin cements are unknown; however, clinicians handling resin cements with altered, setting time (ST) may experience some clinical difficulties [14].

All these factors can affect the durability of restoration and prevalence of caries.

Nevertheless, no previous studies have investigated the physical and mechanical properties of PLS modified resin cements; thus, the aim of this study is to evaluate the effect of incorporating different concentrations of PLS particles into two types of self-adhesive resin cements (RelyXU200 and Breeze) on selected physical and mechanical properties (degree of conversion, compressive strength, film thickness, setting time, water sorption, and solubility).

The null hypothesis of this study is that there is no statistically significant difference regarding the degree of conversion, film thickness, water sorption, solubility, setting time, and compressive strength of the selected resin cements before and after PLS incorporation.

2. Materials and Methods

2.1. Materials. The materials involved in this study, with their compositions, are presented in Table 1.

2.2. PLS Incorporation. Four concentrations of PLS (0.2%, 1%, 2%, and 5%) were selected to be incorporated into each type of resin cements [15]. The method of incorporation started by adding a measured amount of PLS powder weighed using a digital balance (0.1 mg) (AE ADAM AFA-210LC). PLS was added to each resin cement's base and catalyst components and mixed evenly to obtain the required concentration (0.2%, 1%, 2%, and 5%). These percentage was obtained by measuring the weight of powder to the volume of resin cement paste. The base and catalyst were expelled separately then PLS powder was added and mixed evenly using a mixing machine (Zhermack) for 1 min [16].

The mixed paste was then transported into a 5 ml disposable syringe and placed upright on a vibrator to get rid of any trapped air bubbles. Then, each paste was retransferred to its original barrel by pushing the material through a specialized customized connector. Each barrel was left for 5 min to allow the paste to settle. The two barrels were then joined together to be ready for use. All mixing steps were performed in a dark room to avoid unwanted cement polymerization.

2.3. Sample Grouping. The samples of the study were categorized into two main groups depending on the type of resin cement used:

(1) RelyX™ U200 Auto mix Self-Adhesive Resin Cement.
(2) Breeze Self-Adhesive Resin Cement.

Each group was subdivided into five subgroups depending on the PLS concentration (Figure 1).

2.4. Characterization Tests

2.4.1. FTIR and Degree of Conversion. FTIR was performed with two objectives: the first objective was to characterize cement used in this study before and after PLS addition and to find whether PLS incorporation into both resin cement resulted in a chemical reaction. The second objective is to study the degree of conversion of the groups under study.

The DC of the photo-polymerized specimens of all groups was determined using an attenuated total reflection (FTIR–ATR) spectrometer (Shimadzu, USA) with 400–4000 cm^{-1} scanning range at 4 cm^{-1}.

The DC determined from the aliphatic C=C peak at 1,638 cm^{-1} and the aromatic C=C peak at 1,608 cm^{-1} was used for internal calibration. The DC was then calculated by comparing the heights of the peaks for the methacrylate vinyl group in the cured material with those in the uncured

Table 1: Luting cement materials and PLS used in the study.

Material	Description	Composition	Manufacturer	Batch number
RelyX™U200 automix	Self-adhesive dualcure	Methacrylate monomers containing phosphoric acid groups Methacrylate monomers Methacrylate monomers Alkaline (basic) fillers Silanated fillers Initiator components Stabilizers Rheological additives Pigments	(3M ESPE, Germany) 3M Deutschland GmbH	(3M ESPE, Germany) (LOT NO. 8278778, EXP. Date: 03/05/2023) (shade: TR).
Breeze™	Self-adhesive dualcure	Mixture of BISGMAUDMA, TEGDMA, HEMA, and 4-MET resinsSilane treated Barium borosilicate glasses Silica Initiators Stabilizers and UV absorber Organic and/or inorganic pigments Opacifiers	Pentron Clinical Technologies, LLC 68 North Plains Industrial Road Wallingford, CT USA 06492	
Poly-l-lysin		$(C6H12N2O)_n$	Zhengzhou bainafo bioengineering company.Ltd. China	Batch nO 20210901 mfg date 2021.09.20 Shelf life :24 months

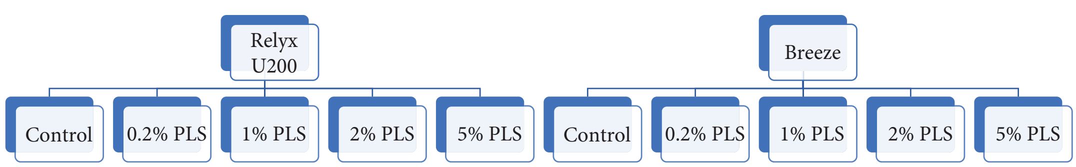

Figure 1: Schematic outline of the sample groups.

material using the formula: [17]

$$DC(\%) = (1 - C/U) \times 100, \quad (1)$$

C and U are the normalized absorption peak heights for the cured and uncured materials.

2.4.2. Compressive Strength. Eighty cylindrical specimens (6 ± 0.1 mm in height and 4 ± 0.1 mm in diameter) were prepared (40 specimens from each cement type). The tip of the automix tube was positioned to one side of the unfilled mold. The mass was extruded slowly to encourage laminar flow. The mold was slightly overfilled with each material and sandwiched between two glass plates under constant pressure with a standard load of 500 mg over the mold to extrude any excess and provide parallel flat specimen [18]. The specimens were light cured for 20 s for RelyX and 40 s for Breeze (according to the manufacturers' instructions), using a light curing device (Eighteeth; China) providing the light intensity of 900 mW/cm^2 at each end of the cylindrical mold. The specimens were then removed from molds and stored in distilled water for 1 day before mechanical testing.

A universal testing machine (Instron model 5569, USA) with a 500 N load cell was used for testing the compressive strength with a cross head speed of 0.5 mm/min. The compressive load was applied to the long axis of each specimen and the maximum load to failure was recorded [19]. The compressive strength, P (MPa), of each individual cylindrical specimen was calculated by dividing the fracture force (F) by the area of the specimen where D was the specimen diametrer, where the compressive determined from Equation (1) [19, 20]

$$P = 4f/\pi D^2, \quad (2)$$

where F was the load at fracture (N) and D was the mean specimen diameter (mm).

2.4.3. Film Thickness. For a luting cement to allow complete seating of the prosthetic restorations, it must obtain an appropriate flow rate maintaining a minimum film thickness. Reduced cement film thickness can also decrease the marginal discrepancies, which in turn reduce the plaque accumulation, periodontal disease, and cement dissolution [21].

A film thickness of the different luting cement groups was performed following ISO 4049:2019(E)

For each group, eight specimens were used. The procedure started by placing 0.01 g of the standard mixed material in the middle of a glass plate [21]. The top glass was placed over the material and subjected to 15 kg (150 N) for 180 ± 10 s. Immediately after the period of loading release the loading system and irradiated the specimens through the center of upper glass plate for twice the recommended exposure time which was recalibrated after each recording then from differences between reading A and reading B film thickness was calculated each sample was measured ten times. Measurements were performed using an electronic gauge (digital micrometer) with an accuracy of 0.5 μm, which was recalibrated after each recording Thus, the film thickness was calculated, and each sample was measured ten times.

2.4.4. Setting Time. Setting time (ST) starts as soon as the redox reaction (mixing the initiator and amine) is initiated and is the time the material takes to reach the limit viscosity, which corresponds to the formation of a solid mass and, consequently, a near complete polymerization [22].

A thermocouple device was used for measuring setting time. A cylindrical mold of 4 mm diameter, 8 mm height with a thermocouple tip protrudes 1 mm into the base of the specimen well. Measurement temperature was maintained at (37 ± 1)°C; the material was mixed and placed inside the mold. The setting time was recorded according to ISO specification 4049:2019 (the test was performed eight times for each subgroup).

2.4.5. Water Sorption (WS) and Solubility (SL). Eight cylindrical specimens were prepared for each subgroup with the dimensions of (15 ± 0.1 mm) diameter and (1 ± 0.1 mm) thickness. The samples were subjected to a desiccation procedure, and then water sorption (WS) and solubility (SL) were measured following ISO specification 4049:2019(E). WS and SL were calculated following gaining $m1$ (constant mass at the beginning), $m2$ (after immersion in water for 7 days), and $m3$ (constant mass after storage) using the following formulae:

$$WS = (m2 - m3)/V \quad (3)$$

$$SL = (m1 - m3)/V, \quad (4)$$

V mean volume of the sample.

2.4.6. Statistical Analysis. The statistical analysis was perfrmed using two-way ANOVA repeated measured test for degree of conversion and two-way Anova for ather tests, Tukey's post hoc complementary test was applied for multiple comparisons between different concentrations. Normality and homogeneity of all testing data were evaluated before ANOVA. Normality was first conducted using Shapiro—Wilk test test, which showed that all groups are normal distribution ($p > 0.05$). For homogeneity evaluation Levene test was used. Levene test showed homogeneity of variance between the groups of each property ($p > 0.05$). These analyses were performed using the IBM SPSS version 26.0 software (IBM Corp., Chicago, IL, USA), and statistical significance was evaluated at the level of 0.05.

3. Results

The FTIR of PLS incorporated cement revealed no emergence of new peaks and no change in the original peaks. This indicated that no chemical reaction had occurred due to the addition. PLS also contained similar functional groups present in RelyXU200 and Breeze. The FTIR spectra for PLS showed a broad medium absorption band at 3,244 cm^{-1} assigned to the NH–CO on the 3,200 B-sheet. The characteristic peaks for NH_2 formation were evident at 3,429 cm^{-1}. While CH_2 asymmetrical and symmetrical peaks appear on 3,074 cm^{-1} and 2,935 cm^{-1}, respectively, C=O stretching appears at 1,674 cm^{-1}, NH at 1,564 cm^{-1}, CH at 1,392 cm^{-1}, and CN appear at 1,255 cm^{-1} [23].

FTIR spectra showed several important peacks for RelyxU200 (1,728, 1,610, 1,1031, and 3,360 cm^{--1}), and Breeze (1,728, 1,612, 1,064, and 3,390 cm^{--1})[1] corresponding to functional C=O, C=C, C–O–C, and O–H/N–H groups, respectively (Figures 2, 3 and Table 2). The results of all ISO tests coincide with ISO standardized limit.

The results regarding (degree of conversion, film thickness, water sorption, solubility, and setting time) were significantly lower for RelyxU200 related to Breeze ($p = 0.001$). On the other hand, the compressive strength was significantly higher for Relyx compared to Breeze ($p = 0.001$). PLS addition on both types of cement has no significant change on all tests except for 5% ($p < 0.05$) in Tables 3–5.

4. Discussion

In indirect restorations such as crowns, inlays, onlays, and bridges, cementation is essential. Since cement employed for adhesion between tooth structure and the internal surface of the restoration are primarily responsible for long-term clinical success. They play a vital role in the cementation [24] because residual bacteria beneath restorations and plaque formation around restoration margins are considered key factors for indirect restorative failure; luting cement with antibacterial properties is desirable. This study focused on the effect of adding PLS on the mechanical and physical properties of two types of luting cements. However, additional study is required to clarify the antibacterial performance of these cements after PLS addition.

FTIR spectra of both types of cement displayed several significant peaks where RelyXU200 had (1,728, 1,610, 1,031, and 3,360 cm^{--1}) while Breeze had (1,728, 1,612, 1,064, and 3,390 cm^{--1})[1] corresponding to their related functional groups (C=O, C=C, O–H, N–H) respectively.

Also, the FTIR spectrum of PLS demonstrated broadband (3,257 cm^{-1}) assigned to the amine group, (3,082 and 2,931 cm^{--1}) belonged to the asymmetrical and symmetrical aliphatic (CH_2) group, respectively. Besides (1,672 and 1,560 cm^{--1}) is a good indicator for stretching (C=O and NH), respectively.

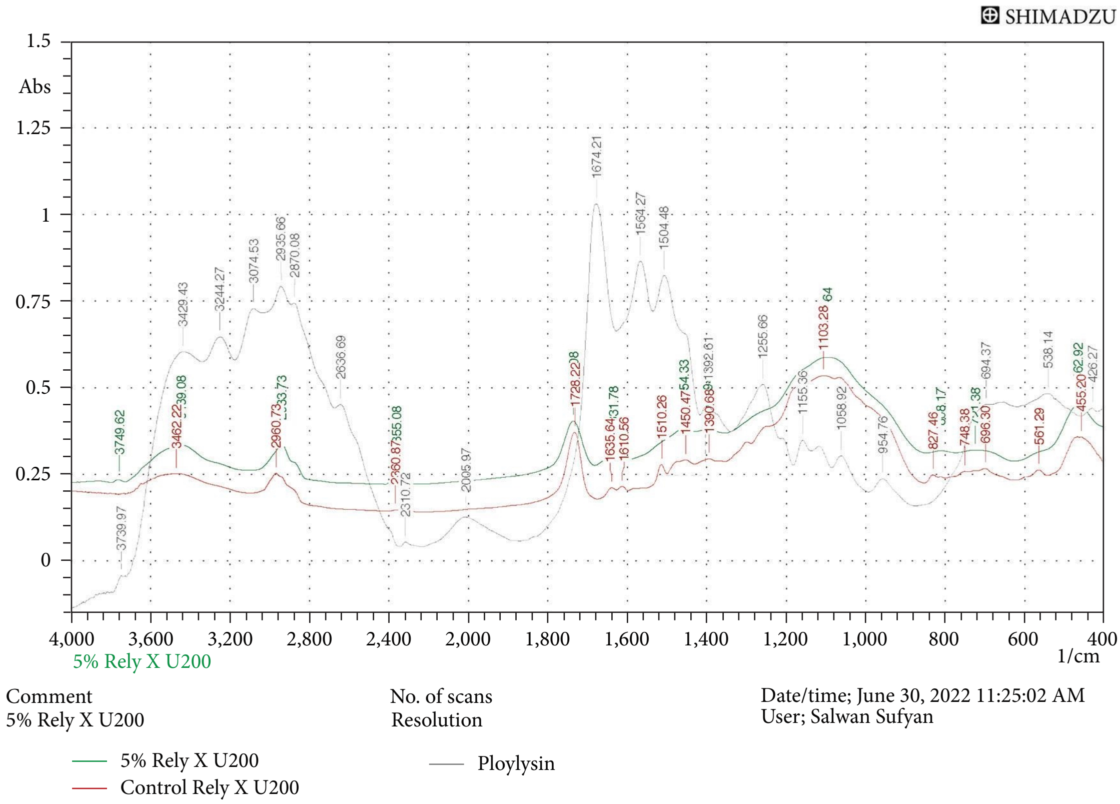

FIGURE 2: The FTIR spectrum of the PLS, control RelyXU200, and 5% PLS incorporated RelyXU200.

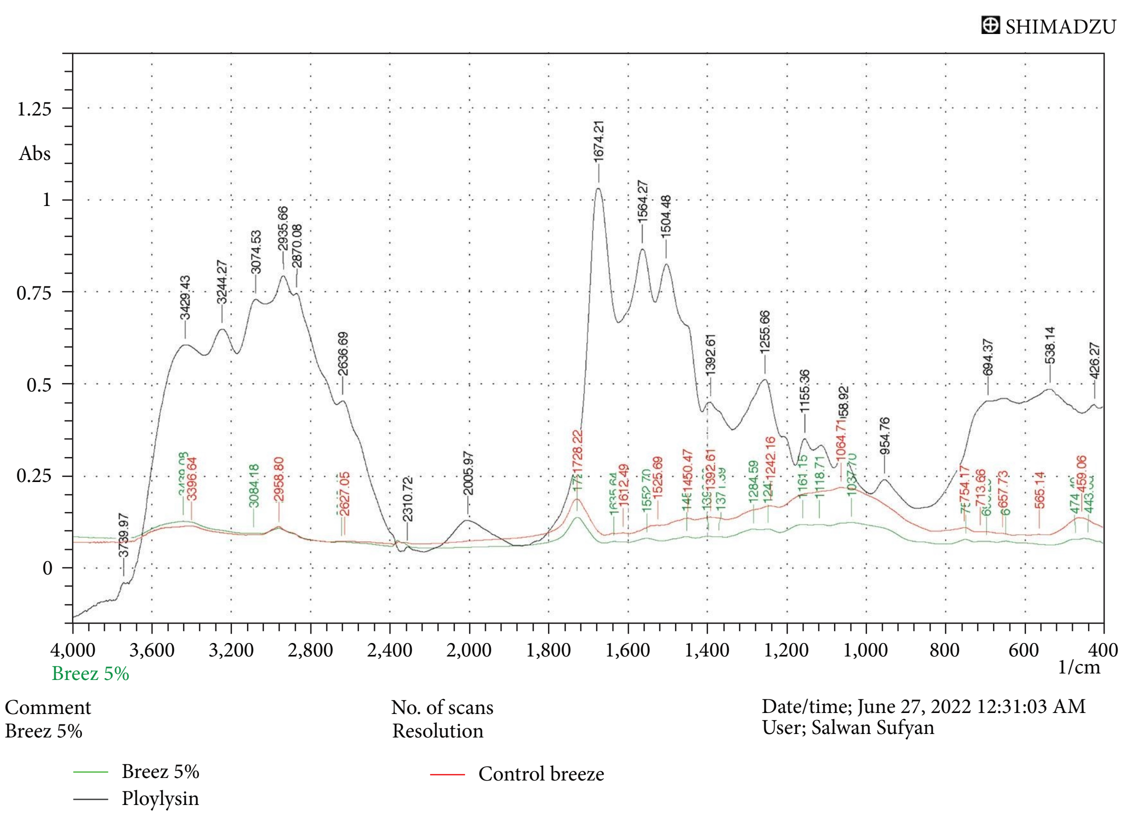

FIGURE 3: The FTIR spectrum of the PLS, control Breez, and 5% PLS incorporated Breeze.

TABLE 2: Infrared bands for poly-L-lysine, RelyxU200, and Breeze.

Wave number (cm^{-1})	Notes
Polylysine	
3,244	NH–CO, amide
3,429	NH_2
3,074	asymmetrical CH_2
2,935	CH_2 symmetrical
1,674	C=O stretching amide I
1,564	CN
1,392	CH, aliphatic
1,255	CN
Relyx and Breeze	
1,728	C=O amideI
1,610, 1,612	C=C
1,131, 1,064	C–O–C
3,360, 3,390	O–H/N–H

According to the result of FTIR, it is evident that no new peaks have emerged after the addition of PLS, and no change has occurred for the present peaks. This indicates that no chemical reaction has occurred after the addition of PLS to both types of cement. Moreover, the peaks of functional groups of PLS and modified cement lie on the same wave number Field [25]. The study's findings revealed that Breeze has a significantly higher degree of conversion than RelyXU200. This finding could be related to the following:

(1) Breeze contains diluent comonomers (HEMA and 4-META). The comonomers (HEMA and 4-META) could boost hydrophilicity and wetting power. These elements support increasing conversion by allowing the diluent monomer HEMA to react with the remaining C=C bonds of the di- and multifunctional monomers that become trapped in the set polymer. These elements will lower the resin matrix's glass transition temperature and raise the mobility of reactive monomers, improving monomer conversion [26].

(2) Additionally, the presence of UDMA in the Breeze composition improves the ultimate conversion in the appropriate polymers as UDMA molecules increase molecular mobility [27].

(3) The quantity of free radicals formed during irradiation. Therefore, the irradiation period plays a crucial role in this process. Breeze 40 s curing period, in contrast to RelyXU200 20s curing time, may have accelerated polymerization and raised the degree of conversion.

Values of the degree of conversion after 24 hr had increased significantly from immediate curing. This could be related to the postpolymerization or dark cure, which occurs in adhesive monomers during the first 24 hr postcuring. This is because the free radicals and unreacted monomers remain trapped within the resin matrix shortly after the start of light curing. Thus, upon ceasing light irradiation, a slow generation of free radicals continues to occur [28].

According to this study, adding PLS has no significant effect on DC% of both types of resin cements except 5% Breeze which causes a substantial decrease in the degree of conversion.

This could be explained by the fact that organic components primarily influence the degree of conversion and are minimally influenced by filler parts (PLS is considered a filler part) in resin cement and do not affect the degree of conversion.

An increasing quantity of PLS may have an adverse effect on DC%. Adding PLS at a high concentration resulted in decreasing the DC%. This may be attributed to a considerable increase in viscosity. It is well known that a polymerizing medium's initial viscosity impacts the polymerization kinetics and may cause premature gelation, which lowers the final degrees of conversion [24].

The mechanical and physical characteristics of luting cement throughout the restoration will determine how well the repair performs clinically. These materials will be subjected to mastication pressures and transfer stresses from indirect restorations to the tooth structure. Therefore, luting cement must possess greater strength to retain the durability and success of restorations [29].

The result of this study revealed that the compressive strength of RelyXU200 was significantly higher than that of Breeze. This result may be due to differences in cement composition (resins and fillers) [24]. Although the whole view (picture) of the composition of Relyx is missing, little information has been collected from previous studies demonstrating that RelyXU200 contains Bis-GMA and TEGDMA. However, Breeze contains additional resin monomers (including HEMA and UDMA).

HEMA, when uncured, absorbs water; this can lead to monomer dilution and thereby halt the polymerization process. Following polymerization, poly-HEMA also attracts water and produces hydrogels, reducing the polymer's mechanical strength [30]. According to the study's findings, PLS addition had no discernible effect on either cement's compressive strength, except resin cement, which included 5% PLS.

The possible explanation for the above results could be related to the fact that when the fillers are incorporated in high concentration, the thickness of the matrix between two fillers is thin enough to create a local stress concentration. The distance between the fillers may be small enough, if not nonexistent, to not behave like a viscoelastic material but like an elastic network [31].

Five percent could be regarded as high enough to cause such an adverse effect. PLS fillers were added without any salination process. Therefore, enhancement of mechanical properties is not expected since such enhancement necessitates the presence of a chemical bond between the resin matrix and filler via a coupling agent [32].

Since a low-film thickness will improve restoration seating and prevent marginal leakage and loss of marginal integrity, reducing plaque accumulation, periodontal disease, and secondary caries, the film thickness is clinically significant and crucial for the clinician. Numerous elements, including consistency, filler content, resin composition, degree of polymerization, and setting reaction, impact cement film thickness

Table 3: Means and standard deviation of the degree of conversion.

	RelyXU200 immediate	RelyU200 24 hr	Breeze immediate	Breeze 24 hr
Control	57.533 (± 0.897)a	62.785 (± 0.775)b	68.628 (± 0.483)Ac	71.167 (± 0.664) Bd
0.2%	57.508 (± 1.131)a	62.774 (± 0.402)b	68.331 (± 0.476)Ac	71.937 (± 0.834)Bd
1%	57.518 (± 1.332)a	62.750 (± 0.847)b	68.005 (± 0.701)Ac	71.660 (± 0.917)Bd
2%	57.894 (± 1.167)a	62.760 (± 0.939)b	67.430 (± 0.506)Ac	71.403 (± 0.808)Bd
5%	57.082 (± 0.566)a	62.321 (± 0.738)b	59.197 (± 1.704)Cc	67.738 (± 0.930)Dd

Note: Different letters (lowercase in the horizontal and uppercase in the vertical) show significant differences ($p > 0.05$) in Tukey's complementary test.

Table 4: Mean and standard deviation of compressive strength (compressive strength, film thickness, setting time, and water sorption).

	Compressive strength		Film thickness		Setting time		Water sorption	
	RelyXU200	Breeze	RelyXU200	Breeze	RelyXU200	Breeze	RelyxU200	Breeze
Control	198.000 (±8.318)Aa	156.733 (± 3.482)Ab	22.333 (± 1.366)Aa	29.333 (± 1.633)Ab	2.167Aa (± 0.126)	3.469ABb (± 0.321)	23.075 (± 1.344) Aa	34.851 (± 0.655)Ab
0.2%	193.183 (± 9.677)Aa	145.600 (± 2.143) Ab	24.000 (± 1.265)Aa	31.333 (± 3.502) Ab	2.167Aa (± 0.126)	3.323Ab (± 0.276)	23.113 (± 0.822) Aa	35.224 (± 1.640)Ab
1%	191.167 (± 14.440)Aa	141.050 (± 18.717) ABb	23.000 (± 2.098)A a	29.667 (± 2.582)Ab	2.000Aa (± 0.178)	3.521ABb (± 0.274)	23.895 (± 0.989) Aa	35.568 (± 1.511)Ab
2%	190.283 (± 6.749)Aa	142.267 (± 3.096)ABb	25.000 (± 1.549)Aa	32.000 (± 3.521)Ab	1.917Aa (± 0.199)	3.625Bb (± 0.118)	23.960 (± 0.967) Aa	35.989 (± 0.860)Ab
5%	158.167 (± 7.387)Ba	128.717 (± 5.340) Bb	30.667 (± 2.422)Ba	38.667 (± 5.715)Bb	1.271Ba (± 0.177)	4.021Cb (± 0.1D88)	27.578 (± 1.195) Ba	38.266 (± 1.459)Bb

Note: Different letters (lowercase in the horizontal and uppercase in the vertical) show significant differences ($p < 0.05$) in Tukey's complementary test.

Table 5: Means and standard deviation of water solubility.

	RelyXU200	Breeze
Control	1.355 (± 0.433)Aa	3.223 (± 0.453)Ab
0.2%	1.415 (± 0.315)Aa	3.618 (± 0.666)Ab
1%	1.498 (± 0.595)Aa	3.521 (± 0.402)Ab
2%	1.6939 (± 0.391)Aa	3.736 (± 0.480)Ab
5%	3.200 (± 0.360)Ba	4.569 (± 0.598)Db

Note: Different letters (lowercase in the horizontal and uppercase in the vertical) show significant differences ($p \leq 0.05$) in Tukey's complementary test.

[32]. This study showed that film thickness for all studied groups passed the requirement of ISO standardization (40 μm) according to ISO 4049-2019.

The result showed that the film thickness of RelyXU200 is significantly lower than that of Breeze. This result may be due to the low viscosity of RelyXU200, which is thought to be influenced by the amount of filler, the kind of filler particles, the composition of the resin matrix, and the degree of polymerization [33]. Breeze filler percentage by volume (52%) Breeze while in RelyXU200 filler percentage by volume is less than that of Breeze (43%), so this may lead to the film thickness of RelyXU200 being lower than that of Breeze [34, 35].

There was a nonsignificant rise in film thickness for both types of resin cement with different concentrations of PLS except for 5%. This may be due to adding a small amount of unbonded filler, which leads to a nonsignificant increase in film thickness, while 5% PLS increases film thickness. The highly filled resin cement tends to flow less, forming a thicker film thickness. The result of this study showed that the setting time of Breeze was significantly longer than that of RelyXU200. The setting time of both types of cement has passed the ISO setting time (Breeze was 3.469 min) and (RelyXU200 2.167 min), which is less than the ISO maximum setting time) of 10 min.

Although little information in the literature is available regarding the composition of both types of cement, it is evident from the available data that Breeze contains calcium and barium minerals, which are not included in the RelyXU200 component list. These minerals act to prolong the setting time owing to their positively charged ions that act by increasing the setting time and prolonging the maturation reaction. This is due to larger cationic ratios or possibly the mixture of cations with various sizes released during cross-linking network development [36].

Another possible explanation is the presence of calcium/strontium ions in Breeze, which form complexes with initiators/activators, acting as inhibitors, thereby affecting the curing time and reaction rate [16]. This study showed that adding PLS to either cement has no significant effect on setting time. This may be related to the small amount of PLS added to both cements. Five percent PLS addition causes a substantial change in the setting time for both cements (decrease the setting time for RelyxU200 and increase it for Breeze).

This may be because PLS has a dual action. The first action is as a secondary amine activator [17]. This is true for Relyx (amine-free initiator). The second action more expected to be worked with a breeze is that PLS acts as a cation, thus increasing the cationic ratio and affecting the development of a cross-linking network. That may cause the setting time to be delayed [36].

Since the luting material is still in communication with the oral cavity on the margin and with water from dentin, the water sorption and solubility characteristics of the luting material are particularly essential [15].

The highest water sorption standard defined by the International Standards Organization is 40 g/mm^3, while the maximum solubility standard is 7.5 g/mm^3.

This study's water sorption and solubility result met the ISO specification since these data were less than the maximum allowed ISO standard (ISO 4049-2019).

It has also revealed that Breeze has substantially higher water sorption and solubility than RelyXU200. This may be related to the higher hydrophilicity of Breeze than RelyXU200. Higher hydrophilicity may be attributed to many components, such as (C-HEMA, UDMA, and META) [37, 38].

According to this study, the addition of PLS for both types of cement has no significant effect regarding water sorption except for the 5% samples.

The possible explanation for that is that PLS concentration is deficient; therefore, their effect is negligible, while with a 5% concentration of PLS, the amount was increased, so the PLS' hydrophilic characteristic encourages water sorption, which raises the mass and volume [39].

Due to the amine group (RNH2+), which converts polylysine into a cationic-charged hydrophilic polymer, PLS promoted significant water-sorption and higher solubility.

This result was confirmed by Khan [16], Liaqat [30], Lygidakis [38], Panpisut et al. [39], and Katsimpali [40].

According to the results of this study, the null hypothesis was accepted since no differences were statistically found in the selected characterization tests for both cements before and after PLS addition, except for 5% concentration.

The main limitation of this study is that it is performed in vitro, with its incomplete ability to capture the inherent complexity exsisting in the oral cavity. Factors related to humidity, temperature and temperature changes, salivary composition and flow, all these might have an impact on the study results. For this reason, further analysis, especially clinical assessment with long term follow-up is required.

5. Conclusions

Within the limitations of this in vitro study, the following conclusions were drawn:

(1) Both self-adhesive resin cement' mechanical and physical properties have not been adversely affected by the PLS addition and have passed the ISO standardization.

(2) Incorporating antibacterial particles into resin cement could be a promising clinical step, through their release at indirect restoration–tooth margin preventing secondary caries initiation.

Ideally resin cement should not be toxic to the pulp cells, and surrounding oral tissues. In the future biocompatibility of the new formulations of resin cement is required. Clinical trials with longer observation periods are required to confirm the data collected from this study.

Data Availability

The data used to support this study are available from the corresponding author upon request.

Ethical Approval

Ethical approval (ref. number: 502-10/3/2022) was obtained from the Research ethics committee of the College of dentistry, university of Baghdad.

Disclosure

The lead author Zainab M Mansi stated that this manuscript is an honest, accurate, and transparent account of the study being reported; there are no important aspects of the study have been omitted.

Conflicts of Interest

The author(s) declare that they have no conflicts of interest.

Authors' Contributions

Conceptualization, writing—original draft preparation, and investigation are done by ZM and AM; methodology is done by ZM; writing—review and editing is done by AM. All authors have read and agreed to the published version of the manuscript.

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