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Comparative Evaluation of Compressive Strength and Flexural Strength of Innovative Nanozirconia and Nanodiamond Filled Novel Resin Cement

Aaqil Arshad Hulikatti and Saleem D. Makandar *

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Comparative Evaluation of Compressive Strength and Flexural Strength of Innovative Nanozirconia and Nanodiamond Filled Novel Resin Cement

Aaqil Arshad Hulikatti and Saleem D. Makandar *

School of Dental Sciences, Universiti Sains Malaysia, Health Campus, 16150 Kota Bharu, Kelantan, Malaysia

* Correspondence: saleemdm@usm.my

Abstract

Self-adhesive dual-cure resin cements (DCRC) simplified clinical application to a single-step procedure. Studies reported inferior mechanical properties compared to conventional resin cements. This study evaluated and compared the compressive strength (CS) and flexural strength (FS) of commercial DCRC against its modification using 10 vol% nanozirconia and 10 vol% nanodiamond. Three groups were prepared: Group 1 (commercial resin cement), Group 2 (nanozirconia-modified), and Group 3 (nanodiamond-modified), with 10 samples per group. 3-(Trimethoxysilyl) propyl methacrylate was used as coupling agent. Specimens were prepared according to manufacturer instructions and tested for CS and FS using a Universal Instron testing machine. Data was analysed using one-way ANOVA and Tukey's post hoc test. Compressive strength values were Group 2 = 132.18 ± 27.93 MPa, Group 3 = 126.21 ± 12.54 MPa, Group 1 = 121.12 ± 19.35 MPa. Flexural strength values were Group 2 = 72.5 ± 10.4 MPa, Group 3 = 71.06 ± 6.3 MPa, Group 1 = 66.92 ± 5.27 MPa. Both nanozirconia and nanodiamond incorporation showed improvements in CS and FS compared to the control group. Within the limitations of this study, nanozirconia modified dual cure resin cement showed higher values compared to nanodiamond modified dual cure resin. These results support further research to optimize nanofiller-reinforced luting cements.

Keywords: nano-modification for cement; nanozirconia; nanodiamond; dual-cure resin cement; self-adhesive resin cement; mechanical properties of resin cement

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1. Introduction

Ceramic restorations are contemporary restorations widely used in patients with high esthetic demands for correction of minor interdental spacing, shape and color of the teeth, particularly the anterior teeth. The success of such restorations depends mainly on the durability of the bond strength between tooth structure and ceramic substrate which is achieved by using luting cements [1-4]. Luting cements are essential to bond the indirect restorations with the tooth surface. Traditionally zinc phosphate was used for cementation for more than a century and hence it is considered as gold standard against which all other cements are tested and compared [5–7]. Other luting cements include zinc polycarboxylate, glass ionomer cement and the latest being the resin cements. Today resin cements are the materials of choice over other luting cements due to their superior properties such as resistance to wear, low solubility, high retention, versatility and long lasting and good aesthetics [8]. Also they are biocompatible, have excellent optical properties and are repairable [9–11]. Resin cements were introduced in dentistry in 1970s and since then have been the material of interest and focus among the dental practitioners and researchers [12,13].

Resin cements are composite materials with less viscosity and consist of fillers and initiators that facilitate lower film thickness along with adequate working and setting time [14]. Dual-cure resin cement is preferred resin cements due to high degree of conversion. N. Hofmann et al found that when dual-cure resin cement was only self-cured (without light exposure), there was a steep decline

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in its strengths, flexural strength by 68.9%, elasticity by 59.2% and hardness by 86.1% compared to the one cured both by light and self-cure [15]. They are used for cementation of full cast-metal crowns, ceramic crowns, zirconia constructions, indirect composite restorations, traditional metal–ceramic prosthesis, metal and glass fiber post, core build-up, implant-supported crowns and bridges, ceramic veneers, orthodontic braces and so on [16–20]. They basically consist of an organic resin matrix, inorganic filler particles and a coupling agent that bonds them together.

There are various types of resin cements developed over years, each with some uniqueness. Earlier resin cements consisted of polymethyl methacrylate which underwent high polymerization shrinkage after curing and it's compressive strength (CS) and flexural strengths (FS) were too low and to overcome this, the number of methacrylates was reduced with addition of filler particles [21]. Contemporary resin cements use dimethacrylate monomers like bisphenol-A-glycidyl methacrylate (BisGMA), bisphenol-A-ethoxy dimethacrylate (BisEMA) and urethane dimethacrylate (UDMA) [22]. Of these monomers, BisGMA is the most used due to its high strength and hardness [13]. These monomers cause polymerization shrinkage due to their high viscosity therefore diluents like TEGDMA (triethylene glycol dimethacrylate) and EGDMA (ethylene glycol dimethacrylate) are added to reduce viscosity and facilitate increased filler content [23]. If polymerization is incomplete, then it results in fast degradation of resin matrix leading to higher chances of fracture and debonding [24].

Filler particles are added to resin matrix to enhance its properties. Nanoparticles have large surface area and disperse easily in the polymer resin matrix and hence enhance the physical and mechanical properties of the resin cements [25]. Various filler particles were used such as quartz, silica, oxides of zinc and aluminum, calcium silicate, zirconia nanofiller, , hydroxyapatite crystals, barium silicate, strontium silicate etc. [26–29] and all of them have been successful in improving different mechanical properties in varying degree. In this study zirconia and diamond nanoparticles (NPs) are used and their effect on compressive strength and flexural strength is being studied.

ZrO_2 is 96%-99% crystalline because of which it has high flexural strength (>900MPa), fracture toughness, and hardness, good corrosion resistance, superior physio-mechanical and biological properties, satisfactory esthetics, and bio inert with excellent biocompatibility [30,31]. Some of the properties are mentioned in Table 1 [32].

Table 1. Mechanical properties of nanozirconia.

Mechanical qualities	**Value**
Density	6.05g/cm^2
Hard strength	1200HV
Flexural strength	900-1200MPa
Compressive strength	2000MPa
Resistance to fracture	7-10MPam$^{1/2}$
Elastic modulus	210GPa
Thermal expansion co-efficient	11*10^{-6}1/K

Diamond nanoparticles are widely considered as smart nanomaterial because of their extreme hardness (10 on Mohs scale), modulus of elasticity (>1000GPa), thermal conductivity, biocompatibility, thermal expansion, optical transparency, chemical corrosion resistance, excellent tribological properties and electrical insulation [33–36]. The properties of diamond nanoparticles is superior to bulk diamond form [37,38]. Some of the properties are mentioned in Table 2.

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Table 2. Mechanical properties of nanodiamond.

Mechanical qualities	**Value**	**References**
Density	3.515 g cm–^{3}	[39]
Hard strength	56 GPA-257 GPA 10 on Mohs scale	[35,40]
Compressive strength	~54 GPa	[41]
Fracture toughness	**(≈ 8–18 MPa·m^0.5)**	[42]
Young's modulus	1050 GPa	[43]
Thermal expansion co-efficient	≈1.1×10^{-5} K^{-1}	[36]

Improving compressive strength, tensile strength and elastic modulus is the basic purpose of adding these filler particles to the resin matrix [44]. The recommended percentage of filler content in resin cement is between 10 wt% - 30 wt% because increasing the percentage of fillers from 10 wt% up to 70 wt% results in increased material rigidity leading to increased polymerization stresses and failure [45]. When volume percentage is taken into consideration then the filler content should be between 31 vol%- 66 vol% [46] and between 17.36 vol%-53.56 vol% [47]. Filler particles of same size in the material often results in void formation hence incorporating filler particles of different sizes is advisable to improve packing density and the performance of rein cement [48,49]. Studies have found that the use of filler particles, their size and morphology, Silane treatment greatly improvise the strength and reduce the wear and polymerization shrinkage of resin cement [50–56].

A good bond between the organic resin matric and inorganic filler particles is essential for a resin cement to last longer and this bonding is achieved using a binding agent called a coupling agent, usually silanes [57]. Silane has the ability to bond between the hydroxyl groups of inorganic filler particles as well as methacrylate group of organic resin matrix [13] and hydrolysis of Silane agents often jeopardizes the bond strength.

Good luting cement not only establishes a strong bond between tooth structure and the indirect restoration but also possesses excellent physical and mechanical properties to withstand occlusal forces. Bite forces range between 100N-320N [58]. To withstand such a force the resin cement should have high compressive and flexural strength. Some of the causes of the failure of resin cement are microleakage and secondary caries, adhesive, cohesive and mixed failures, polymerization shrinkage and so on. Till date there is no resin cement available that possesses mechanical properties resembling that of ideal resin cement therefore resin cement has been the subject of extensive studies and investigation since their introduction in dentistry. The objective of this study was to compare and evaluate the compressive strength and flexural strength of self-adhesive dual-cure resin cement with its modifications using 10 vol% nanozirconia and 10 vol% nanodiamond filler particles in an effort to obtain novel resin cement with improved mechanical properties.

2. Materials and Methods

The materials used in this study were RelyX U200 dual-cure resin cement (3M ESPE), zirconia oxide nanoparticles (Vedayukt India Pvt Ltd), diamond nanoparticles (Vedayukt India Pvt Ltd) and 3-(Trimethoxysilyl)propyl methacrylate coupling agent (Tokyo Chemical Industry (India) Pvt Ltd) and is mentioned in Table 3. Three groups were prepared using these materials. The groups were as follows. Group 1: Commercial dual-cure resin cement, group 2: 10% Nanozirconia modified dual-cure resin cement, group 3: 10% Nanodiamond modified dual-cure resin cement. These groups are mentioned in Table 4. Nanozirconia and nanodiamond, each were added 10% volume to commercially available dual-cure resin cement RelyX U200. The volume was measured by using graduated test tube. Two different moulds made of clear acrylic were procured for testing compressive strength and flexural strength. The measurements of the moulds are as follows. Compressive strength moulds of size of 6mm height x 3mm diameter. Flexural strength moulds of size 25mm length x 2mm breadth x 2mm height.

Flowchart of the methodology is depicted below in Figure 1.

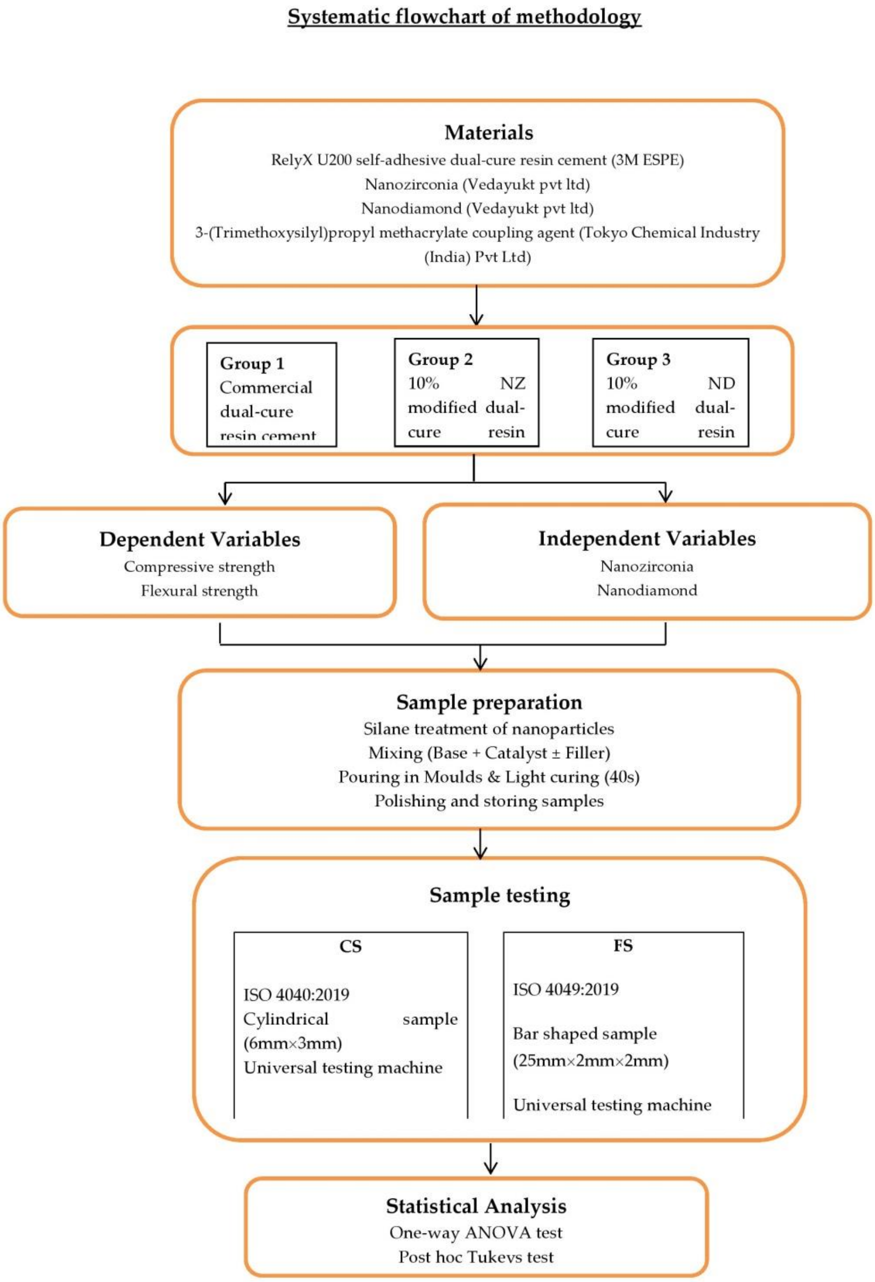

Figure 1. Systematic flowchart of methodology.

One important thing to note here is the use of nanofiller in volume percentage. Different nanoparticles possess different densities which makes volumetric measurement less reliable in cases where absolute weight accuracy is crucial. However, the primary objectives of this study was to compare and evaluate the properties of resin cement by just altering filler type (zirconia and diamond) at specific volume fraction while maintaining other experimental conditions constant. This approach is seen in literature where volume% was used to express nanoparticles addition rather than weight% [59–62].

Secondly, in clinical conditions, resin cements are mixed volumetrically thus it reflects actual mixing conditions. Also that, both the nanofillers followed same protocol of measuring and mixing thus ensuring consistency even if their densities varied.

As for the issue about agglomeration and dispersion control, similar studies are found were nanoparticles were manually dispersed with coupling agent without significantly affecting agglomeration and dispersion [63–65]. And as mentioned earlier, the purpose of this study to comparative evaluation and not for absolute density calibration, so this method is adequate and is reproducible. However, based on the findings of this study it is recommended to further investigate by means of varying methodology and approach.

Table 3. Materials used in the research study.

Material	Brand name	Serial number
RelyX U200 A2 Shade	3M ESPE	30064000 (HSC Code)
Nanozirconia	Vedayukt India Pvt Ltd	1314-23-4 (CAS)
Nanodiamond	Vedayukt India Pvt Ltd	7782-40-3 (CAS)
3-(Trimethoxysilyl) propyl Methacrylate	Tokyo Chemical Industry (India)Pvt Ltd	2530-85-0 (CAS)

Table 4. Groups in the study.

Nanoparticles	Group 1 (control)	Group 2	Group 3
Nanozirconia (vol%)	-	10 %	-
Nanodiamond (vol%)	-	-	10%

Group 1- 11g of 100% RelyX U200 dual-cure resin cement was used as the control group. The base and catalyst were measured in small increments in equal quantities using digital weighing machine (Precision balance, LWL Germany, Model: LB-210S) and mixed manually over the glass slab to obtain a uniform mix. This mix was then loaded thoroughly in the moulds, avoiding air bubbles and voids formation. Then each sample was light cured for 40 seconds to obtain a well cured sample using Woodpecker i-LED Plus curing light whose light intensity at normal mode is approximately 1000–1200 mW/cm². The tip of curing light was placed closely over the sample and the sample was cured on both sides. Total 20 samples were prepared with 10 samples each for compressive strength and flexural strength testing. The samples were then removed from the moulds and polished at room temperature by using silicon carbide sandpapers of 1000, 1200, 1500 and 2000 grit. The samples were marked and stored at room temperature.

Group 2- 10% nanozirconia particles were mixed with coupling agent on a glass slab. This mixture was then added to base paste of commercial dual-cure resin cement (RelyX U200) taken on another glass slab. This base paste and the catalyst paste were then measured in small increments in equal quantity using digital weighing machine (Precision balance, LWL Germany, Model: LB-210S)

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and were mixed well on the glass slab and loaded in the moulds, avoiding air bubbles and voids formation. Then each sample was light cured for 40 seconds to obtain a well cured sample. Samples were prepared with 10 samples each for compressive strength and flexural strength testing. The samples were then removed from the moulds and polished at room temperature by using silicon carbide sandpapers of 1000, 1200, 1500 and 2000 grit. The samples were marked and stored at room temperature.

Group 3- Commercially available dual-cure resin cement (RelyX U200) was taken 90% by volume which is equivalent to 1067mg and 10% by volume nanodiamond was measured using a graduated test tube. Similar process as that of group 2 preparation was followed to prepare group 3 using 10% nanodiamond particles by volume and the samples were stored at room temperature.

The volume percentage to weight percentage for nanozirconia and nanodiamond is mentioned below in Table 5 and Table 6 below.

Table 5. Volume% to Weight% for nanozirconia.

Material	**Volume%**	**Weight mg**	**Weight%**
RelyX U200	90%	978	88.90%
Nano zirconia	10%	122	11.10%

Table 6. Volume% to Weight % for nanodiamond.

Material	**Volume%**	**Weight mg**	**Weight%**
RelyX U200	90%	1067mg	97%
Nano diamond	10%	33mg	3%

Adding nanoparticles to base paste while maintaining manufacturer's recommendation of base: catalyst 1:1 by weight is commonly accepted methodology, though it dilutes the monomer when used at higher concentrations, however at lower concentration, as in this study, this affect is negligible and this was confirmed by FTIR analysis in earlier studies [65,66]. Alteration of initiator and accelerators in the catalyst paste affects the chemistry and introduces greater variability and reduces clinical relevance but here since variation is in the nanofiller and base paste so it does not affect or alter the polymerization stoichiometry. We acknowledge that FTIR-based DC analysis was not performed and recommend it as a limitation and direction for future research.

Compressive strength was measured according to ISO 4049/2019. The samples were cylindrical measuring 6mm height and 3mm diameter. The samples were placed vertically in the loading frame of computerized and software based universal instron testing machine (ACME Engineers, India Model No. UNITEST-10) and held securely using grips (Figure 2 a&b) and load was gradually applied till the samples broke. The speed of the machine was 1mm/minute. The load at which the samples broke was recorded.

Flexural strength was measured according to ISO 4049/2019. The samples were placed horizontally in the loading frame of the universal instron testing machine and held securely with grips and load was gradually applied at the head speed of 1mm/minute till the samples broke. The load measuring cell records the load applied. The results were analysed using one-way ANOVA with post-hoc Tukey HSD Test.

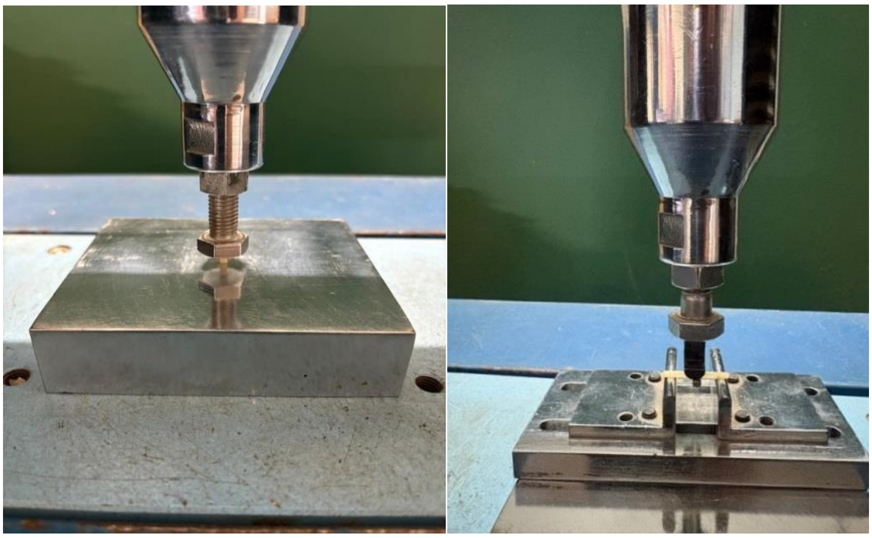

Figure 2. a) Testing CS, b) Testing FS.

3. Results

There was no statistically crucial difference in compressive strength among the groups (p = 0.505). Group II (Nano zirconia) had the highest mean compressive strength (132.18 ± 27.93 MPa), followed by Group III (Nano diamond) at 126.21 ± 12.54 MPa, and Group I (commercial cement) at 121.12 ± 19.35 MPa. While the trend suggests a slight improvement with Nano reinforcement, the statistical analysis does not support a meaningful difference between the groups.

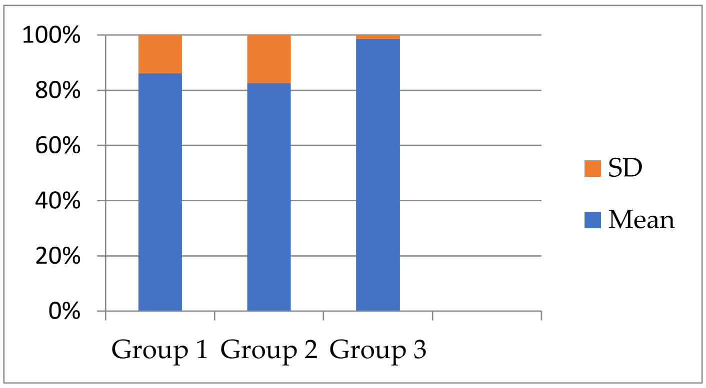

Figure 3. Comparison of compressive strength values of One-Way ANOVA test of all 3 groups.

Table 7. Mean compressive strength values and one-way ANOVA values.

Variable	**Group1 (Mean±SD)**	**Group2 (Mean± SD)**	**Group3 (Mean±SD)**	**F (df_1, df_2)**	**p-value**
Compressive strength	121.12±19.35	132.18± 27.93	126.21±12.54	F(2,27)=0.701	0.505

Table 8. Post Hoc Multiple Comparison (Tukey HSD Test) values of CS.

Dependent Variable	Comparison Groups	Mean Difference (I–J)	Standard Error	p-value	95% CI Lower	95% CI Upper
Compressive Strength	Group 1 vs. Group 2	-11.060	9.353	0.473	-34.229	12.109
	Group 1 vs. Group 3	-5.973	9.353	0.850	-29.143	17.196
	Group 2 vs. Group 3	5.087	9.353	0.910	-18.083	28.257

Insignificant differences were observed between the groups for compressive strength ($p > 0.05$), suggesting that the addition of nanofiller did not meaningfully alter the compressive properties of the resin cement.

There was no statistically prominent difference in flexural strength among the three groups ($p = 0.256$). Group II (Nano zirconia) had the maximum mean flexural strength (72.50 ± 10.40 MPa), followed closely by Group III (Nano diamond) at 71.06 ± 6.30 MPa, and Group I (unmodified resin cement) at 66.92 ± 5.27 MPa. Although the modified groups demonstrated numerically higher values, the lack of statistical significance indicates that these differences may be due to random variation and not the effect of nanoparticle modification.

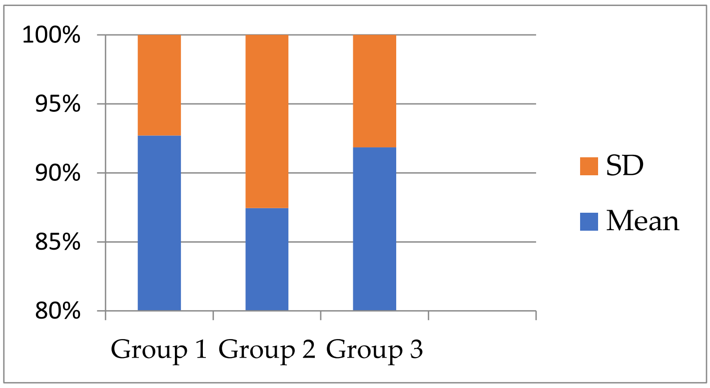

Figure 4. Comparison of flexural strength values of One-Way ANOVA test of all 3 groups.

Table 9. Mean flexural strength values and one-way ANOVA values.

Variable	Group1 (Mean± SD)	Group2 (Mean± SD)	Group3 (Mean± SD)	F (df_1, df_2)	p-value
Flexural strength	66.92 ± 5.27	72.50 ± 10.40	71.06 ± 6.30	F(2, 27) = 1.435	0.256

Table 10. Post Hoc Multiple Comparison (Tukey HSD Test) values of flexural strength.

Dependent Variable	Comparison Groups	Mean Difference (I–J)	Standard Error	p-value	95% CI Lower	95% CI Upper
Flexural Strength	Group 1 vs. Group 2	-5.580	3.420	0.250	-14.061	2.901
	Group 1 vs. Group 3	-4.144	3.420	0.457	-12.625	4.337
	Group 2 vs. Group 3	1.436	3.420	0.909	-7.045	9.917

Insignificant variations were noted between any of the groups ($p > 0.05$), indicating that the type of resin cement had no notable impact on flexural strength.

4. Discussion

Compressive strength: Compressive strength is defined as the maximum compressive stress that a material can withstand before failure [67]. It is also the capacity of a material to resist a load that tends to reduce its size [68]. CS is a very critical factor in success of the materials since high CS is essential to withstand masticatory and Para-functional stresses [69–71]. In this study Compressive strength of group 1, group 2 and group 3 was found to be 121.12 ± 19.35 MPa, 132.18 ± 27.93 MPa, 126.21 ± 12.54 MPa, respectively. Amongst the three group, the CS of commercially available dual-cure resin cement was lowest followed by that of the resin cement modified with 10% nanodiamond filler and the highest CS was found with resin cement modified with 10% nanozirconia fillers. Though there was a slight improvement in the CS of both the modified resin cements compared to the RelyX U200 but the change was very minor and insignificant.

The compressive strength of zirconia nanoparticles is 2000 MPa [32] as a result they are the material of high interest in various fields and have proven to be highly efficient and useful. They have been used as filler particles in various dental cements, including resin cements, and the outcome has been promising in improving the mechanical properties of the cements. Current study focuses on the effect of zirconia nanoparticles on CS of resin luting cement and the results are not satisfactory as there is only a minor improvement in CS of resin cement. This is contrary to the findings of a previous study in which significant improvement in CS was noted and higher CS was noted with higher the percentage of zirconia NPs [72]. To the best of our knowledge, there is no previous study conducted using nanodiamond to evaluate their effect on compressive strength of luting resin cement. Though diamond nanoparticles are a hard and strong material so their addition was expected to enhance CS of resin cement but in this study we found very minimal improvement hence first null hypothesis was partially rejected.

Flexural strength: The flexural strength of a material is the maximum stress that it can resist before failure when subjected to bending load [73]. Flexural strength of Yttrium stabilized zirconia polycrystal (Y-TZP) is >900MPa [74]. The flexural strengths obtained in this study for group 1, group 2 and group 3 are 66.92 ± 5.27 MPa, 72.50 ± 10.40 MPa, 71.06 ± 6.30 MPa, respectively. The results were similar to that of compressive strength, that is, though there was a minor improvement in the flexural strength of experimental groups compared to control group but the change was insignificant.

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The results of this study are in agreement with that of the previous study conducted wherein it was found that the addition of zirconia nanoparticles partially improved FS of resin cement and the change was insignificant [75]. It also agrees with the studies conducted earlier wherein it was found that the type and loading of filler particles affects the FS [76–78]. One more finding in this study, when compared to the study conducted by Raja Azman Raja Awang et al. is that the percentage of zirconia nanoparticles added is directly proportional to the FS[79], but increasing it also increases the rigidity of the resin cement [47] which is undesirable for a luting cement, though it is essential for restorative resins wherein a very high FS is more desirable [80–84]. This study, however, is in conflict with the findings of other studies wherein incorporation of zirconia NPs was found to enhance the flexural strength of resin matrix [85–87].Based on the findings of this study, second null hypothesis is partially rejected as there is no significant change in the CS values of experimental resin cements.

Similarly, the addition of diamond nanoparticles also partially enhanced the flexural strength of the resin cement. Though there is no previous study on the effect of nanodiamond (ND) particles on flexural strength of resin luting cements but there is a study conducted on their effect on polymethyl methacrylate (PMMA) denture base resins and it was found that NDs enhance the FS of denture base resin [88]. In that study it was found that addition of low concentration of ND (0.1% and 0.25%) had higher FS compared to that of higher concentration of NDs (0.5%). Similar findings were observed in another study conducted on denture base resins [89]. These studies could possibly explain the reason for marginal improvement in the FS values with addition to a much higher percentage of NDs (10%) in this study, though further investigation is warranted on this subject for confirmation.

Table 11 summarizes the gist of the study, its findings, advantages and disadvantages, limitations and comparison of its results with previous studies to analyze whether they are in agreement or in conflict.

Table 11. Summary of discussion.

Properties	Findings of Current Study	Advantages / Positive Outcomes	Disadvantages / Negative Outcomes	Risks / Limitations	Comparison with Previous Studies
Compressive Strength (CS)	Group 1 (Control): 121.12 ± 19.35 MPa Group 2 (10% Nano-ZrO_2): 132.18 ± 27.93 MPa Group 3 (10% Nano-Diamond): 126.21 ± 12.54 MPa $p = 0.505$ (NS)	•Slight increase in CS in both modified groups compared to control. •Supports possibility of reinforcement.	•No statistically significant difference between groups ($p > 0.05$). •Limited mechanical benefit.	•High NP (10 vol%) may cause particle agglomeration, affecting homogeneity and polymerization. •Possible interference with base-catalyst ratio altering curing kinetics.	•**Contradicts** prior study [56], which found significant improvement in CS with higher NZ addition. •**Novelty:** First study testing nanodiamond in resin luting cement hence no prior reference available for direct comparison.

Flexural Strength (FS)	Group 1: 66.92 ± 5.27 MPa Group 2: 72.50 ± 10.40 MPa Group 3: 71.06 ± 6.30 MPa p = 0.256 (NS)	•Minor increase in FS in both modified groups, showing potential for reinforcement. •Confirms filler addition does not adversely affect cement integrity.	•Statistically insignificant improvement (p > 0.05). •High filler content may increase stiffness without significant strength gain.	•Agglomeration at 10 vol% may reduce efficiency of stress transfer. •Higher rigidity is unsuitable for luting cements as it requires some elasticity.	•**Contradicts** studies [69–71] reporting significant enhancement in FS with ZrO_2 NPs. •**Consistent** with PMMA studies [72,73], where low ND concentration improved FS more effectively than higher concentrations.

5. Conclusions

In this study, addition of 10 vol% nanozirconia and nanodiamond filler particles to dual-cure resin cement did not have statistically significant improvement in both compressive strength and flexural strength compared to unmodified resin cement (p>0.05). Zirconia reinforced dual cure resin cement group showed higher values than nanodiamond modified dual cure resin cement group, which was followed by unmodified resin cement. This suggests that, within the limits of this study, though mechanical properties under the study were not significantly affected but even a slight positive enhancement indicates potential for reinforcement thus encouraging further investigation with different concentrations of nanoparticles, different approach and methodology to obtain a notable improvement in the mechanical properties.

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Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

BisGMA	bisphenol-A-ethoxy dimethacrylate
BisEMA	bisphenol-A-ethoxy dimethacrylate
UDMA	urethane dimethacrylate
CS	Compressive strength
FS	Flexural Strength
DCRC	Dual cure resin cement

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