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SEM-EDS and FTIR Analysis of Innovative Nanozirconia and Nanodiamond Filled Novel Resin Cement

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Article

SEM-EDS and FTIR Analysis of Innovative Nanozirconia and Nanodiamond Filled Novel Resin Cement

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Abstract

Success of indirect restorations highly depends on the adhesive cement used. In the last couple of decades resin cement has evolved greatly and is preferred over other cements. It is important to understand the chemical composition and their interaction with teeth and the restoration in order to improvise and overcome the disadvantages. Here three groups were prepared, RelyX U200 dual-cure resin cement as control group or group 1and 10 vol% nanozirconia and 10 vol% nanodiamond modified cement as group 2 and group 3, respectively. 3-(Trimethoxysilyl) propyl methacrylate was the coupling agent used. Nanoparticles were added to coupling agent and mixed well on a glass slab, this was then mixed with the base of the commercial resin cement to obtain a homogenous mixture. Upon setting, the samples were crushed in mortar using pestle and made fine powder and incubated at 37^{0}C and 100% humidity for 24 hours followed by SEM-EDS (SEM-EDS, Quanta FEG 450 USM) analysis. For FTIR analysis the powder form was made into the KBr pellet in a 1:10 ratio using samples and potassium bromide. The pellet was further analysed for chemical composition.

Keywords: nanozirconia; nanodiamond; resin cement; FTIR; SEM EDS; dual-cure resin cement

1. Introduction

Restoration of teeth is essential for a healthy oral cavity in particular and overall health of an individual in general. This restoration can be either direct in the form of fillings or indirect restorations such as crowns and bridges, inlays, onlays, posts, core build up, veneers and so on. Indirect restorations require a good cement to bond them firmly with the tooth surface. Over last few decades adhesive cements has evolved greatly and there has been tremendous achievement in improving the biomechanical properties of these cements. Various cements are used for cementation such as zinc phosphate, zinc-oxide eugenol, zinc polycarboxylate, glass ionomer cement and the latest is resin cement. They were first introduced in dentistry in 1970s and since then have undergone a lot of changes and today different types of resin cements are available.

Generally, resin cements consist of an organic resin matrix, inorganic filler particles and these two are held together and bonded by a coupling agent [1]. Silane bonds between the functional groups (eg- hydroxyl, methoxy) of inorganic filler particles as well as unsaturated organic groups (methacrylate group) of organic resin matrix [2,3]. Stronger the bond between the resin matrix and the filler particles better will be the cement. A weak bond is often due to hydrolysis of the coupling agent [4] therefore a hydrophobic coupling agent is preferred [5]. Resin cements are preferred because they are biocompatible [6], have better aesthetics and adhesion [7], good marginal adaptation [8], better mechanical and physical properties (flexural strength and elastic modulus) compared to other cements [9], low solubility [10,11], superior bond strength [12] and long lasting . Like other luting cements, resin cements too have their own shortcomings. Some of the problems with resin cements are polymerization shrinkage, staining, post-operative sensitivity, microleakage, staining, fracture etc. They are used for cementation of full-cast metal crowns, ceramic crowns, zirconia crowns

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and bridges, indirect composite restorations, traditional metal–ceramic prosthesis, metal and glass fibre-post, core build-up, implant-supported crowns and bridges, and ceramic veneers [13]. A successful restoration is dependent not only on the tooth preparation and the restoration design but also on the adhesive cement used. A restoration with good tooth preparation and correct design might also fail if the resin cement being used fails, which could be due to reasons such as polymerization shrinkage[14,15], micro-fracture of resin cement [16], plastic deformation of resin cement [17,18], incomplete polymerization of resin cement[19,20] and hydrolysis of coupling agent [21].

Failures of restoration can be broadly categorized as cohesive, adhesive and mixed failures. Cohesive failures are because of failures related to resin cements. Hence it is very important to understand the chemistry of the resin cements in order to predict their interaction with the tooth structure and the restoration and understand their bonding mechanism to further improvise their adhesive property [22]. The most common analysing methods in doing so are scanning electron microscopy along with energy dispersive x-ray spectroscopy (SEM-EDX) and fourier transform infrared spectroscopy (FTIR). SEM use is indicated in investigating the surface morphology, topography and irregularities of materials such as minerals, alloys, ceramics, polymers, glasses, powders, etc. [23–28]. SEM is a unique method of gathering information a material's surface by means of high-resolution images. It also creates a three-dimensional image by combining targeted beam of ions and scanning electron based analysis technique. A focused beam of electrons is used to scan the surface of a specimen and generate images at a much greater resolution compared to optical microscopy [29]. In this technique, fine powder of the sample is coated most commonly with gold. Palladium can also be used. This coating is ultrathin and is deposited using low vacuum sputter or by high vacuum evaporation [30]. This prevents artefacts of the images. The artefacts could be formed in non-conducting samples which gets charged when exposed to electron beam so the coating converts the sample into conducting one. According to Saghiri et al [31], there are 2 functions of these coatings. Firstly to enhance signal and surface resolution especially for samples with low atomic numbers and secondly, resolution is improved due to enhanced emission of backscattering and secondary electrons near the surface as a result a much clear image of surface is obtained. EDX on the other hand is a qualitative and quantitative X-ray micro analytical technique used in chemical analysis to understand the elemental composition and quantitative components of samples [32]. It utilizes characteristic X-rays emitted by the sample during SEM imaging [33]. It is quick, accurate and does analysis on micron scale without destructive effects. FTIR helps in studying the chemical structure, bond strength of the specimens and is an accurate, fast and convenient approach of quantitative analysis. It works by scanning the sample and exploring chemical properties by means of infrared light. It works on the principle of absorption of IR radiation as most of the organic and inorganic components are active in IR range so when the rays are absorbed the photon from lower energy orbit moves to the higher energy orbit, resulting in vibration of the molecular bond [34]. It generates a spectrum of infrared absorption that produces sample's profile which is similar to the exclusive fingerprint of the molecule [35].

Another important aspect of good cement is its inorganic component which provides strength to the cement in withstanding against active functional forces and prevents its failure. Traditionally, fillers like silica, quartz, fine glass particles, hydroxyapatite crystals etc. have been used for a long time but the search for new fillers, which could perform better than previous fillers and also overcome their shortcomings, is underway. Off late, zirconia and diamond nanoparticles as fillers have been of great interest among research professionals and clinicians due their excellent biomechanical properties. Studies have shown promising results with nanozirconia use in resin cement [36–38]. Similarly, nanodiamond has been tested with PMMA denture base resin and polymer composites and has significantly improved its mechanical properties [39–41].

In order to have a long lasting and successful restoration the understanding of the adhesive cements, its chemical nature, its interaction with the teeth and the restorations and its behavioural changes upon addition of inorganic filler particles is of great importance.

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). 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. 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.

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.

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) and were mixed well on the glass slab and loaded in the moulds, avoiding air bubbles and voids formation.

Group 3- Commercially available dual-cure resin cement (RelyX U200) was taken 90% by volume which is equivalent to 1067mg and 10% by volume nanodiamiond 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.

Analysis by SEM-EDX technique-

After the initial set of all the groups, samples were d crushed with pestle and mortar into fine powder and incubated at 37^{0}C and 100% humidity for 24 hours in an incubator. The fine powder of the samples was coated with gold metal. After this process, samples are examined under an electron microscope at 1000x, 5000x, 10,000x and 50000x magnifications followed by EDS analysis ((SEM-EDS, Quanta FEG 450 USM).

Analysis by FTIR technique-

For FTIR analysis, same powder form sample was made into KBr pellet in a 1:10 ratio using samples and potassium bromide. The pellet was further analysed for chemical composition.

Test.

3. Results

Group 1

Figure 1. group 1 image at 10000x.

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Table 1. Analysis of group 2 at 10000x SEM magnification.

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	33.511	22.200
8	O	Oxygen	41.310	36.047
13	Al	Aluminum	4.773	7.100
14	Si	Silicon	14.780	22.900
38	Sr	Strontium	1.614	7.800
40	Zr	Zirconium	4.012	3.953

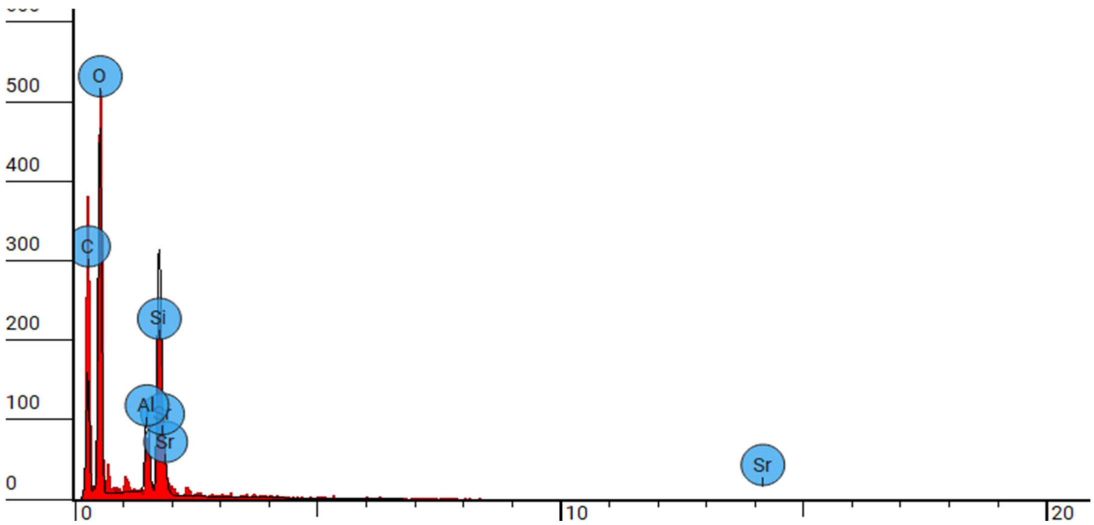

Figure 2. SEM-EDX analysis of group 2 at 10000x.

Group 2

Figure 3. group 2 image at 10000x.

FW: 52 µm, Mode: 10 kV - Low, WD: 9.1 mm, Detector: BSD Full, Time: 3/13/25 4:07 PM

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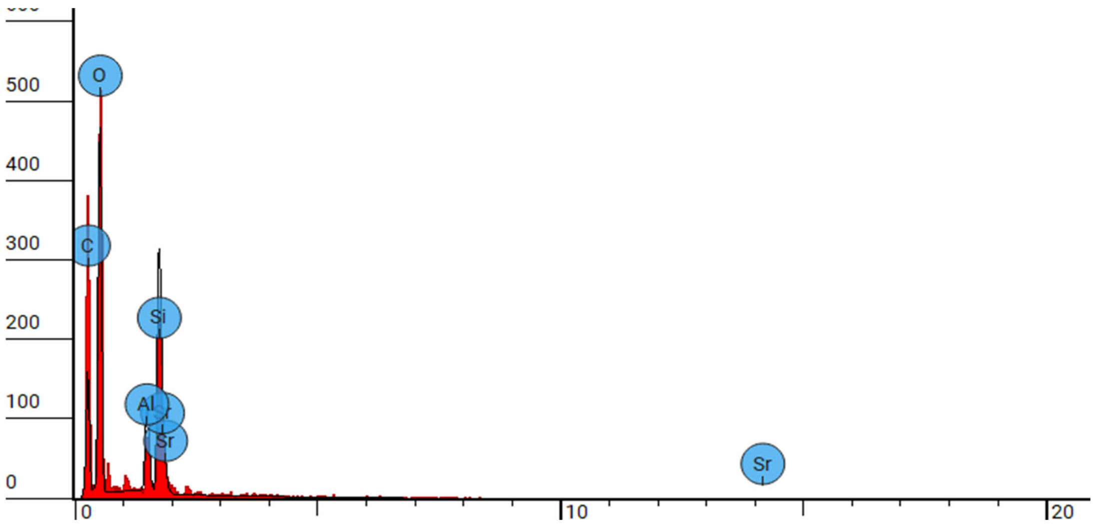

10 769 counts in 0:00:30 (356 c/s)

Figure 4. SEM-EDX analysis of group 2 at 10000x.

Group 3

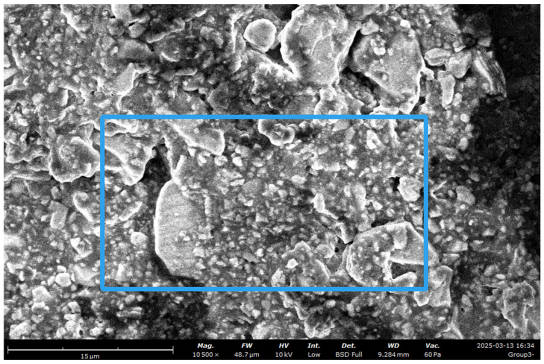

Figure 5. Group 3 image at 10000x.

Table 3. Analysis of group 3 at 10000x SEM magnification.

	Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
	6	C	Carbon	31.654	22.178
	8	O	Oxygen	54.263	50.649
	13	Al	Aluminum	3.810	5.994
	14	Si	Silicon	9.023	14.785
	38	Sr	Strontium	1.251	6.394

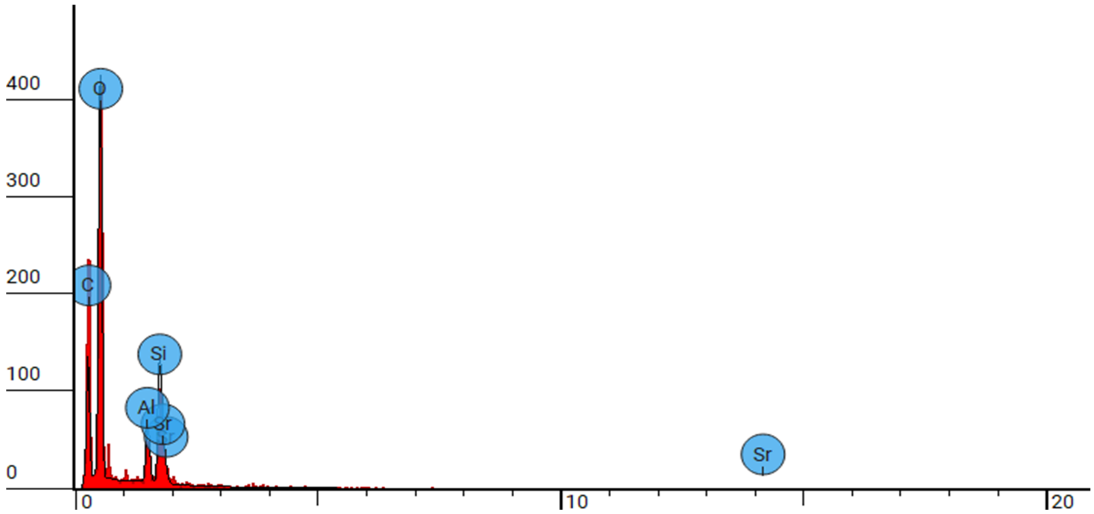

Figure 6. SEM-EDX analysis of group 1 at 5000x.

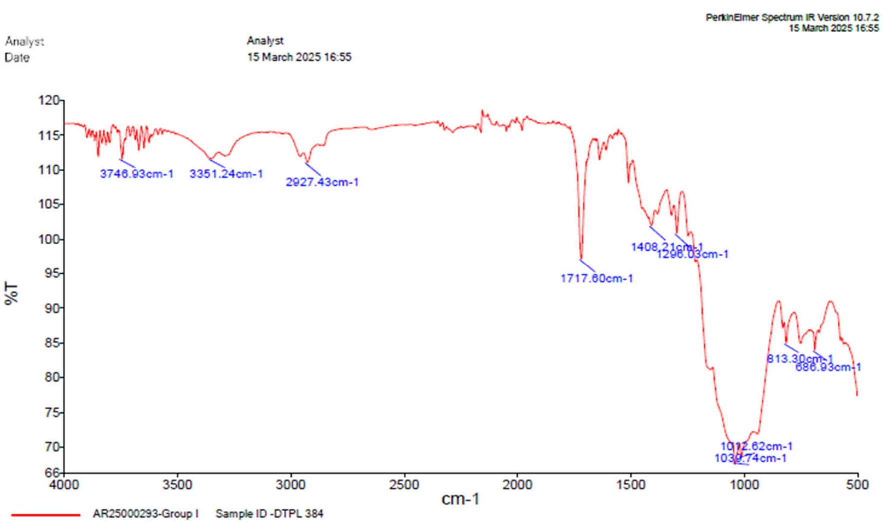

Figure 7. FTIR analysis of group 1.

Table 4. Table depicting absorption range.

GROUP 1			
Absorption (cm^{-1})	**Group**	**Compound**	**Comments**
3672.93	O-H stretching	alcohol	Medium bond, free
3351.24	O-H stretching	alcohol	Strong, intermolecular bonded
2927.43	C-H stretching	alkane	Medium
1717.60	C=O stretching	α, β, unsaturated ester	Strong
1637.69	C=C stretching	Alkene	Monosubstituted
1509.77	N-O Stretching	Nitro compound	Strong
1408.21	O-H bending	Carboxylic acid	Medium
1296.03	C-O stretching	Aromatic ecter	Strong

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1039.74	S=O stretching	Sulfoxide	Strong
1042.62	S=O stretching	Sulfoxide	Strong
813.30	C=C stretching	Alkene	Medium, trisubstituted
686.93	C-I stretching	Halo compound	Strong.

Group 2:

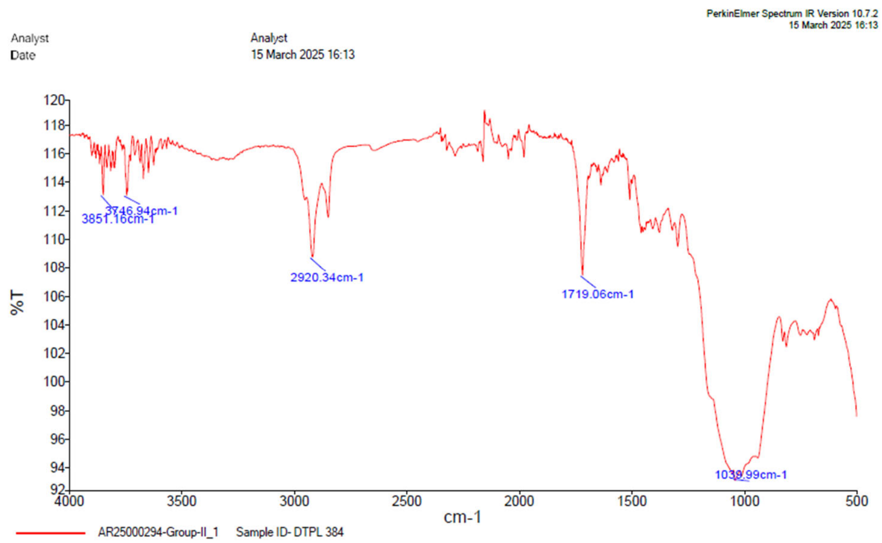

Figure 8. FTIR analysis of group 2.

Table 5. Table depicting absorption range.

GROUP 2			
Absorption (cm^{-1})	**Group**	**Compound**	**Comments**
3851.16	O-H stretching	alcohol	Medium bond, free
3746.94	O-H stretching	alcohol	Medium bond, free
2920.34	C-H stretching	alkane	Medium
1719.06	C=O stretching	α,β, unsaturated ester	Strong
1039.99	S=O stretching	Sulfoxide	Strong

Group 3

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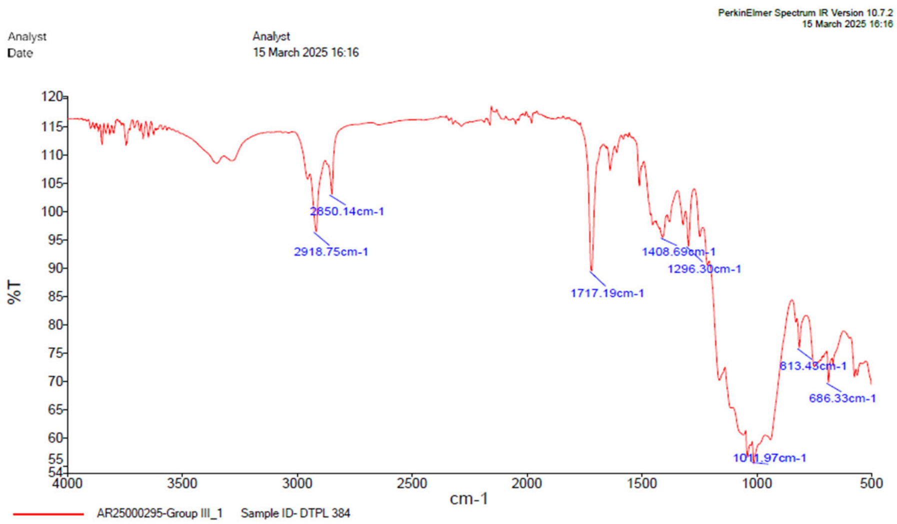

Figure 9. FTIR analysis of group 2.

Table 6. Table depicting absorption range.

GROUP 3			
Absorption (cm⁻¹)	**Group**	**Compound**	**Comments**
2918.75	C-H stretching	alkane	Medium
1717.19	C=O stretching	α,β, unsaturated ester	Strong
1408.69	O-H bending	Carboxylic acid	Medium
1296.30	C-O stretching	Aromatic ester	Strong
1011.97	S=O stretching	Sulfoxide	Strong
813.43	C=C stretching	Alkene	Medium, trisubstituted
686.32	C-I stretching	Halo compound	Strong.

4. Discussion

SEM-EDX:

SEM images captured at varying magnifications provide a detailed depiction of the surface topography and microstructural features of dual-curable resin cement RelyX U200 and different groups of the same type, nanozirconia modified dual-cure resin cement and nanodiamond dual-cure resin cement, highlighting on its physical attributes and potential clinical applications. The SEM images of group 1-3 (DCRC and modified DCRC) depicted in Figures 1,3,5 unveil a heterogeneous surface topography characterized by the presence of diverse particle structures at magnifications of 1000x, 5000x, 10,000x, 50000x. Resin cement exhibits intricate morphological features, comprising a combination of splintered, heterogeneous and round shaped particles distributed in a size range of 1-10 μm. This amalgamation of particulate structures imparts DCRC with a unique surface texture, which could potentially influence its mechanical properties and bonding characteristics. [36,37].

Results from EDS spectroscopy analysis of the dual-curable resin cement RelyX U200 and different groups of similar type, nanozirconia modified dual-cured resin cement and nanodiamond dual-cured resin cement showed a variety in composition and the added fillers. To delineate the restorations to improve mechanical properties, nanozirconia was added. The disadvantage of adding large percentages of radiopaque fillers is the chemical degradation caused by water immersion. It is

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important to balance the mechanical properties of DCRC with the incorporation of filler particles without affecting its physical properties.

Fillers were of varying sizes and SEM findings present diverse filler shapes. The modified cement contained range of spherical shape of fillers to clusters, loosely agglomerated. These particles increased the combined mechanical strength with high biocompatibility of modified DCRC[33,37,43].

Elemental analysis conducted via SEM-EDS reveals the elemental composition of DCRC, confirming the presence of silica, oxygen, aluminium, Phosphorus, Strontium, zirconium and carbon as evidenced by the elemental peaks observed in the backscattered electron images Figures 2, 4, 6. These elemental constituents play pivotal roles in dictating the physico-mechanical properties and clinical performance of DCRC, thereby underscoring their significance in dental material science. These congruent observations validate the robustness and reliability of SEM analysis as a tool for characterizing dental resin cements and elucidating their structural attributes. Moreover, the identification of splintered and round particles within dual-curable cement RelyX U200 and different groups of its type, nanozirconia modified dual-cure resin cement and nanodiamond dual-cure resin cement corroborates previous research findings, further consolidating our understanding of its microstructural architecture and surface characteristics [Figure 1-6]. These consistent observations underscore the reproducibility and inherent characteristics of dual-cure resin cement RelyX U200 and different groups of its kind, nanozirconia modified dual-cure resin cement and nanodiamond dual-cure resin cement, positioning it as a promising candidate for various clinical applications in restorative dentistry.[30,33,43]

FTIR:

Resin cement used in restorative dentistry must be of good mechanical properties and should strongly bind the tooth and the restoration. It is desirable to know the composition, chemical structures and bond strength between different components of the resin cement, as they are placed close to the vital tissues. This is a vibrational technique used to understand the chemical ingredient and structure. Here photons move from lower energy state to higher state by absorbing energy in infra-red region resulting in vibrating movement of molecular bond [38,43].

The FTIR analysis conducted in this study offers a comprehensive understanding of dual-curing resin cement RelyX U200 and different groups of its type; nanozirconia modified dual-cure resin cement and nanodiamond dual-cure resin cement, generating ideas on their molecular structure and bond strength. Characteristic absorption bands corresponding to sulfoxide, alkane and hydroxyl groups were observed in the FTIR spectra of RelyX U200 and different groups of dual cure resin cement, nanozirconia modified dual curable resin cement and nanodiamond double curing resin cement, confirming its composition.[36]

Comparison of the FTIR spectra between dual-cure resin cement RelyX U200 and nanozirconia modified dual-cure resin cement counterpart revealed notable shifts in absorption bands but under same band values indicating interactions between the DCRC components and nanozirconia particles. Particularly, the shifting of the OH stretching vibration from 3672.93cm-1 to 3851.16 cm-1 in the modified formulations suggests alterations in the hydrogen bonding environment, potentially enhancing material properties, [Figure 7-9]. The presence of free functional groups for bonding in certain modified groups highlights the versatility and potential applications of nanozirconia-modified DCRC in dental procedures.[35,36]

Comparison of the FTIR spectra between dual cure resin cement RelyX U200 and nanodiamond modified dual-cure resin cement counterpart revealed notable shifts in absorption bands indicating interactions between the DCRC components and nanodiamond particles with medium bonding. Particularly, the shifting of the OH stretching vibration from 3672.93cm-1 to C-H stretching vibration 2918.75 cm-1 in the modified formulations suggests alterations in the hydrogen bonding environment, potentially enhancing materials properties. The presence of free functional groups for bonding in certain modified groups highlights the versatility and potential applications of nanodiamond-modified DCRC in dental procedures [35,43].

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Similarly, the absorption bands associated with C=C and S=O stretching vibrations in the 2000–1650 cm-1 range provide insights into the presence of aromatic ring structures within the DCRC. These functional groups contribute to the DCRC chemical reactivity and mechanical properties, influencing their suitability for dental applications. Moreover, the assignment of absorption peaks to a broader peak with an idealized formula provides valuable insights into the overall composition of the dual-cure resin cement RelyX U200 and different groups of dual-cure resin cement, nanozirconia modified dual-cure resin cement, nanodiamond dual cure resin cement.[34,43]

5. Conclusions

SEM-EDX findings presents varying shapes of nanoparticles distributed evenly in modified resin cements and in FTIR, molecules were seen distributed evenly with varying bond strength among molecules ranging from medium to strong.

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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
FTIR	Fourier Transform Infrared Spectroscopy
SEM	Scanning electron microscope
EDS	Energy-Dispersive X-ray spectroscopy
DCRC	Dual cure resin cement

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