INTERNATIONAL JOURNAL OF DENTAL MATERIALS VOLUME 2 NUMBER 2 APRIL - MAY 2020

ISSN: 2582 - 2209 (ONLINE)

INTERNATIONAL JOURNAL OF DENTAL MATERIALS

VOL. 2 NO. 2 APRIL - MAY 2020

International Journal of Dental Materials

Volume 2 Number 2 April—May 2020

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Focus and Scope

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Publication Information: International Journal of Dental Materials 2020, volume 2, issue 2 is scheduled for publication. Further details or information is available on this journal's website (https://ijdm.co.in/index.php/dental-materials/).

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Original Article International Journal of Dental Materials 2020; 2(2) ISSN:2582-2209

Comparative evaluation of linear dimensional change and resistance to the compressibility of three polyvinyl siloxane interocclusal recording materials: an *in-vitro* study

Deshpande Prachitee V.[1,*], Wadkar Arti P.[2]

[1]Postgraduate Student, [2]Professor, Department of Prosthodontics, Nair Hospital Dental College, Mumbai, Maharashtra, India.

INFORMATION

Article History

Received *10th April 2020*
Received revised
15th May 2020
Accepted *17th May 2020*
Available online
31st May 2020

KEYWORDS

Bite registration

Polyvinyl siloxane bite registration

Dimensional changes

Resistance to compression

ABSTRACT

Background: Simulation of patient's occlusion on an articulator marks the basis for initiating any prosthodontic treatment. An understanding of the inherent properties of materials used to record the maxillomandibular relationship becomes essential to minimise errors. The soft recording material initially fills the spaces between teeth, hardens, and records the specific relationship of the arches. Elastomers as interocclusal record materials have consistently yielded the least error among the materials studied.

Aim: To comparatively evaluate linear dimensional change and resistance to compression of three polyvinyl siloxane interocclusal recording materials after setting.

Materials and methods: Clonebite (Ultradent), Colorbite D (Zhermack) and Imprint bite (3M ESPE) were evaluated for linear dimensional changes using Travelling Microscope and resistance to compressibility using Universal Testing Machine.

Results: Maximum linear dimensional change was observed in Imprint bite along with the highest resistance to compressibility. The minimum linear dimensional change was seen in Colorbite D while the least resistance to compressibility was seen in Clonebite.

Conclusion: From the results of the present study, the immediate articulation of casts is recommended after the bite registration using the material tested. However, a delay up to 8 hours could be considered as acceptable for Clonebite and Colorbite D.

1. Introduction

The precise articulation of the patient's cast is a prerequisite for diagnosis and subsequent corrective treatment [1]. Interocclusal record is defined as "A registration of the positional relationship of the opposing teeth or arches; a record of the positional relationship of teeth or jaws to each other" [2]. It forms the prime connecting link between the mouth and an articulator. The first interocclusal registration was made in 1756 by Philip Pfaff [3]. Accurate interocclusal records minimize the need for intraoral adjustments during prosthesis delivery and thus, are essential in providing precisely fabricated restorations to reduce

Correspondence: *Corresponding author Email Address: *prachiteedeshpande@gmail.com*

How to cite this article: Deshpande Prachitee V., Wadkar Arti P. Comparative evaluation of linear dimensional change and resistance to the compressibility of three polyvinyl siloxane interocclusal recording materials: an *in-vitro* study . Int J Dent Mater 2020;2(2): 30-36.
DOI: http://dx.doi.org/10.37983/IJDM.2020.2201

overall treatment time and cost [4].

There are various interocclusal recording materials viz. dental plaster with modifiers, modelling compound, waxes, acrylic resin, and zinc oxide eugenol paste. The accuracy of the fit of recording material on the study or working casts is a critical factor as repositioning the record on the cast could be a source of discrepancy. Elastomers as interocclusal record materials have consistently yielded the least error among the materials studied. These materials are easy to manipulate and offer little or no resistance to closure. They set to a consistency that makes them easy to trim without distortion, and accurately reproduce tooth details. Addition silicone and polyether impression materials were modified by adding plasticizers and catalysts in order to be used as interocclusal recording media [1].

Many studies have evaluated the physical properties and behaviour of these materials. A compressive force is usually exerted on the interocclusal recording materials during articulating procedures that may cause inaccuracy during mounting of casts and distortion during fabrication of restorations. The resistance of these materials to compressive forces is critical because any deformation during the recording and mounting process could result in inaccurate articulation of casts and faulty fabrication of restorations [5]. The linear dimensional accuracy is also accountable for errors after the material sets and during transfer of the records on the articulator. It becomes an important property of the interocclusal recording media in order to avoid any discrepancies between the maxillomandibular registration and mounting of the casts. The objective of this study was to evaluate the linear dimensional change at varying time intervals and resistance to compression of Clonebite (Ultradent), Colorbite D (Zhermack) and Imprint bite (3M ESPE) polyvinyl siloxane interocclusal recording material and to suggest the ideal time for articulation for Clonebite (Ultradent), Colorbite D (Zhermack), and Imprint bite (3M ESPE).

2. Materials and methods

Three commercially available addition polysiliconebased bite registration material. i.e., Clonebite (Ultradent) (Material A), Colorbite D (Zhermack) (Material B), and Imprint bite (3M ESPE) (Material C) with similar composition were selected. An apparatus (Universal testing machine) for the determination of strain in compression (Instron 3345-Screw driven 5 kN capacity) and Travelling Micrometer microscope (WeswoxOptik two-motion microscope, 10x Eyepiece, 0. 01 mm Vernier reading) for determination of linear dimensional change were used. All the experiments were conducted at 23°C and 75% humidity [6].

Maxillary and mandibular dentulous study models were mounted in maximum intercuspation on Hanau semi-adjustable articulator (Figure 1) using Kaldent mounting plaster type II [6]. The mounting was stabilized using non-latex number 19 rubber bands which exerted an even force of 25N on the articulator till the plaster set [5]. The bite was opened by 3mm anteriorly according to incisal pin guidance markings.

Sample preparation was done by injecting the interocclusal recording material using the automix technique on the occlusal surface of mandibular teeth and firmly closing the articulator till the incisal pin touched the incisal table (Figure 2). Each material was allowed to set according to the manufacturer's instructions. Left half (3rd quadrant) of the samples was used to assess the resistance to compressibility while the right half (4th quadrant) was used for assessing linear dimensional change.

2.1 Testing the samples for linear dimensional change

Two fixed reference points were marked on the dentulous study models (buccal cusp tip of 1st premolar and mesiobuccal cusp tip of 1st molar) by applying a drop of composite resin on the cusp tips. The distance between the reference points marked on the interocclusal record was measured on the travelling microscope immediately after the material had set and it was noted as 'L'. This reading was measured on the travelling microscope. Subsequent readings were recorded at time intervals of 1 hour, 8 hours and 24 hours. The values were noted as 'Time 1', 'Time 2', and 'Time 3' respectively (n=40).

The linear dimensional change of the materials was assessed by using the following formula [7];

Linear dimensional change= L-(Time1/Time 2/Time3)

2.2 Testing the samples for resistance to compression

Three fixed reference points on the samples, i. e. centre of the incisal edge of the maxillary central incisor (21), the buccal cusp tip of 1st maxillary premolar (24) and mesiobuccal cusp tip of maxillary 1st molar (26) were marked where the minimum thickness of the specimen was 1mm in posterior teeth and 3mm in anterior teeth [7] and were subjected to a load of 20N [6] after the

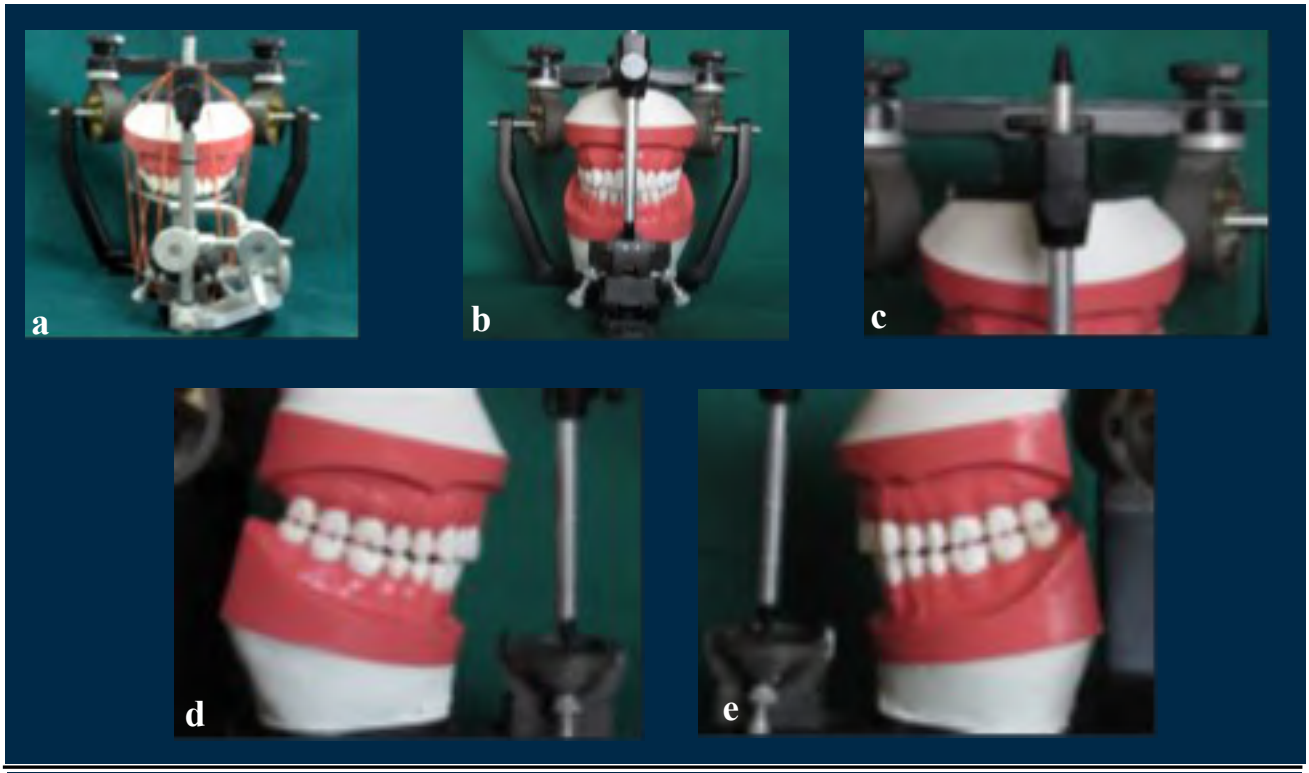

Figure 1. a-e. Mounting of maxillary and mandibular models on a semi adjustable articulator and raising the bite.

Figure 2. Samples prepared with interocclusal materials.

material had set. The strain at this load was recorded as 'X'. The load was brought back to Zero Newtons. Sixty seconds after the application of the first load, the samples were again subjected to a load of 20N [1,7,8]. The strain at this load was recorded as 'Y'. The change in strain in compression was computed as follows;

Change in Strain in compression= (Y-X)/100

2.3 Statistical analysis

Data were subjected to statistical analysis using the Statistical Package for Social Sciences (SPSS v 21.0, IBM). Comparison of numerical values between the groups was done using one-way ANOVA test, followed by Post Hoc Tukey's test for pair wise comparisons. For all the statistical tests, $p<0.05$ was considered to be statistically significant, keeping α error at 5% and β error at 20%, thus giving power to the study as 80%.

3. Results

3.1 Linear dimensional change

Intergroup comparison at 0 hours showed a mean linear dimensional change of Clonebite and Imprint bite to be similar, i.e. 1.32±0.025 and 1.33±0.040 whereas that of Colorbite D to be 1.30±0.35. A comparison at 1 hour, 8 hours and 24 hours showed similar results.

Although significant differences in linear dimensional changes were observed among the materials, the intragroup comparison did not show any statistically significant changes (Table 1).

Linear dimensional change of Clonebite with Colorbite and Imprint bite showed a statistically significant difference at 0 hours. The difference in linear dimensional change between Colorbite D and Imprint bite was not statistically significant. At 1 hour, 8 hours and 24 hours, the comparison between Clonebite, Colorbite D and Imprint bite showed statistically non-significant results (Table 2).

Significant differences in linear dimensional changes were observed between Colorbite D and Imprint bite in the 1st hour. No statistically significant differences were observed between 0-8 hours. All groups showed a statistically significant difference between 0-24 hours; dimensional change of Imprint bite being the highest followed by Clonebite and Colorbite D.

3.2 Resistance to compressibility

Intergroup comparison of at various time intervals and resistance to compressibility at incisor, premolar and molar regions of all three materials showed statistically significant results (Table 3).

In the incisor region, Clonebite and Imprint bite showed a statistically significant difference, with Imprint bite showing higher resistance to compressibility than Clonebite. In premolar region, Clonebite and Imprint bite, as well as Colorbite D and Imprint bite, showed statistically significant differences, with Imprint bite being most resistant to compression in both cases. Results obtained for the molar region were similar to those obtained for the premolar region. Clonebite and Imprint bite, as well as Colorbite D and Imprint bite, showed statistically significant differences in resistance to compression. Imprint bite showed the highest resistance to compression.

4. Discussion

The present study was conducted with a null hypothesis that linear dimensional change and resistance to compression after the setting of three polyvinyl siloxane interocclusal recording materials are not time-dependent entities and do not influence the time of

Table 1: The linear dimensional change in interocclusal materials at different time intervals (Post Hoc analysis - Pair wise comparison)

Time	Groups		Mean Difference	Standard Error	Significance
0 hours	A	B	0.02625*	0.00758	0.002*
	A	C	0.00455	0.00758	0.820#
	B	C	0.03080*	0.00758	0.000*
1 hours	A	B	0.03150	0.02351	0.376#
	A	C	0.01225	0.02351	0.861#
	B	C	0.01925	0.02351	0.692#
8 hours	A	B	0.00950	0.04285	0.973#
	A	C	0.04125	0.04285	0.602#
	B	C	0.05075	0.04285	0.465#
24 hours	A	B	0.01200	0.05045	0.969#
	A	C	0.01575	0.05045	0.948#
	B	C	0.00375	0.05045	0.997#

* Significant difference between the groups.
# No significant difference between the groups.

Table 2. The linear dimensional change in interocclusal materials at different time intervals (Post Hoc analysis - Inter group comparison)

Time	Groups		Mean Difference	Standard Error	Significance
Difference 0-1 hrs	A	B	0.01975	0.00958	0.103#
	A	C	0.01655	0.00958	0.200#
	B	C	0.03630*	0.00958	0.001**
Difference 0-8 hrs	A	B	0.01675	0.01133	0.305#
	A	C	0.01167	0.01133	0.559#
	B	C	0.02842*	0.01133	0.036#
Difference 0-24 hrs	A	B	0.03725*	0.01353	0.019*
	A	C	0.06450*	0.01353	0.000**
	B	C	0.10175*	0.01353	0.000**

* Significant difference between the groups.
# No significant difference between the groups.

Table 3. Resistance to compressibility at incisor, premolar and molar regions of interocclusal materials (Post-Hoc analysis - Pairwise analysis)

Tooth region	Groups		Mean Difference	Standard Error	Significance
Incisor	A	B	7.47450	7.68091	0.595#
	A	C	20.46175*	7.68091	0.024*
	B	C	12.98725	7.68091	0.213#
Premolar	A	B	2.02500	7.29009	0.958#
	A	C	23.36450*	7.29009	0.005**
	B	C	21.33950*	7.29009	0.011*
Molar	A	B	4.47525	6.86386	0.792#
	A	C	26.29175*	6.86386	0.001**
	B	C	21.81650*	6.86386	0.005**

* Significant difference between the groups.
# No significant difference between the groups.

articulation of casts. The above materials were selected for the present study as they are based on polyvinyl siloxane composition, which is widely used in regular clinical practice due to their accurate for registration of the bite and consistent clinical performance.

Michalakis et al. [1] in 2004 stated that both 'material' and 'time' individually as well as in combination, affect the linear dimensional changes. In the present study, Colorbite D showed minimum linear dimensional change indicting comparably higher dimensional stability in

the horizontal plane after setting. Imprint bite, on the other hand, showed maximum distortion within the first hour, indicating least horizontal dimensional stability. Vassilis V. and Tripodakis [9] in 2003 conducted a study in which four bite registration materials were tested. PVS displayed the lowest discrepancy among the tested. When the records were transferred onto the casts, the discrepancies were approximately 0.5 mm, without significant difference among materials. In the present study, statistical differences were obtained in the 1st and 24th hour, indicating variability in the distortion of the materials is time-dependent in spite of same base filler material composition. Tejo S.K. et al. [8] in 2012 conducted a study to assess the dimensional stability of 3 PVS interocclusal recording materials at an interval of 24, 48 and 72 hours. They concluded that Polyvinylsiloxane interocclusal records must be articulated within 24 hours. The present study further shortened this time interval indicating that articulation of casts was done immediately for Imprint bite; however, a delay of up to 8 hours is acceptable for Colorbite D and Clonebite. Breeding L.G., Dixon D. L [5] in 1992, studied the compression resistance of four interocclusal recording materials. Results of one-way ANOVA indicated that there was a significant difference in compressive resistance among the materials of each thickness.

The present study simulates the bite as it is obtained in the patient's mouth, unlike the study as mentioned above, thus reducing the errors in the readings caused due to greater thicknesses which eventually lead to a higher degree in compressibility of the materials. Significantly high resistance to compressibility in incisor, premolar and molar areas was shown by Imprint bite as compared to the other materials. Michalakis et al. [1] in 2004 studied and evaluated the resistance to compressibility after setting of various interocclusal registration materials. The materials used were one polyether bite registration material, four polyvinyl siloxane bite registration materials and one zinc oxide eugenol paste. The test revealed that compared to the rest of the interocclusal materials that were tested, Blu Mousse recorded the highest resistance to compression. Regisil polyvinyl siloxane was the least resistant to compression. In the present study, a force of 20 N was applied and was kept constant throughout the study as compared to a 30 N force applied in this study, which is more than the normal force exerted in the oral cavity during mandibular closure [5]. Out of the three materials tested in the present study, Imprint bite showed the highest resistance to compression in all the three regions, i.e. incisor, premolar and molar regions that were tested indicating highest dimensional stability in the vertical plane. Clonebite showed maximum distortion, indicating a higher incidence of causing vertical discrepancies during articulation procedures. Studies showing testing of these materials have time and again been conducted using a standard steel cylinder method to provide a uniform thickness to the material as per Revised American Dental Association Specification No. 19 For Non-Aqueous, Elastomeric Dental Impression Materials. Such a situation does not exist in the oral cavity, where the bite thickness ranges from 1-3mm.

The present study compared three different polyvinyl siloxane materials of a similar composition for the linear dimensional changes and resistance to compression after setting. Certain limitations were encountered during the same. The properties that were tested in the study are influenced by various other factors like weight changes in the materials, consistency prior to setting etc. The 'Time' factor needed to be further considered as well. Larger sample size should be incorporated for testing. Correlation of dimensional changes occurring in all planes must be made and quantified to get the precise effect of dimensional change occurring along each axis on accuracy in articulation of casts.

5. Conclusion

Within the limitations of this study, the following conclusions were drawn;

a. Colorbite D showed minimum linear dimensional change out of the three materials tested while Imprint bite showed the most indicating that it is most susceptible to distortion in the horizontal plane while stabilising the bite during mounting of casts. Least resistance to compression was shown by Clonebite indicating highest rebound of material in the vertical plane during articulation of casts. Imprint bite showed the least rebound indicating least discrepancies in vertical plane post articulation of casts.
b. The ideal time of articulation of casts for Clonebite, Colorbite D and Imprint bite is ideally immediately when the bite is recorded, i.e. within the 1st hour. However, a delay of up to 8 hours can be considered as acceptable for Clonebite and Colorbite D. A delay in articulation beyond this time should be avoided.

Conflicts of interest: Authors declared no conflicts of interest.

Financial support: None

Acknowledgements: The authors thank Ultradent, Zhermack and 3M ESPE for providing the materials for conducting the study and IIT Bombay for laboratory support and permission to use their testing facilities.

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Review Article International Journal of Dental Materials 2020; 2(2) ISSN:2582-2209

A comprehensive review on electrospinning design, parameters and potential use of electrospun nanofibers in regenerative endodontics

Sai Lakshmi I [1,*], Madhu Varma K[2], Girija S Sajjan[2], Kalyan Satish R[2], Sindhu D[1], Sowmya M[1]

[1]Postgraduate Student, [2]Professor, Department of Conservative Dentistry and Endodontics, Vishnu Dental College, Bhimavaram -534202, West Godavari, Andhra Pradesh, India.

INFORMATION

Article History

Received *3rd April 2020*
Accepted *5th May 2020*
Available online
31st May 2020

KEYWORDS

Electrospinning

Regenerative endodontics

Stem cells

Toxicity

Triple antibiotic paste

Nanofibres

ABSTRACT

Electrospinning is a versatile technique that has gathered interest due to its ability to fabricate nano and microscale fibres with unique properties of high surface area and fibrous porosity. This technique has been widely used in the late 20th (1990) and early 21st (2000) centuries. Since the beginning of its use, significant improvements have been made in the design, materials used, and fibres produced. The electrospinning technique is used to fabricate a material with therapeutic properties as it allows the researchers to incorporate various anti-microbial agents to different polymers without altering the chemical characteristics of polymers.

The production of nanofibres through electrospinning is affected by many operating parameters. It is, therefore, essential to know various parameters and processes that aid in fabricating the desired fibre assemblies. The nanofibres remain an essential division of biomaterials due to a wide range of biomedical applications. Nanofibres have unique properties such as protein absorption, binding sites to cell receptors, can provide maximum volume fraction by controlling fibres' alignment and orientation hence improving the material properties like surface morphology, porosity, and geometry.

Recent trends in endodontics, encourage regenerative therapy for the treatment of necrotic immature permanent teeth for root development and maturation. In this context, efficient disinfection of the root canal system is a crucial step. Existing chemical irrigating solutions (for eg., NaOCl) and antibiotic pastes (for eg., Triple antibiotic paste) usage at higher doses showed toxic results on the pulpal stem cells. Therefore, it was found to be beneficial to use a nanofibre-based intracanal drug delivery construct to release antibiotics at lower, yet anti-microbially effective concentrations.

This review aims to discuss the basic concepts of electrospinning and its potential application in regenerative endodontics along with various parameters, which affect the fibre morphology and properties of produced nanofibres.

1. Introduction

Electrospinning is initially known as electrostatic spinning because it makes use of electrostatic force for the process of spinning. This spinning was first

Correspondence: *Corresponding author Email Address: lakshmiindukuri111@gmail.com

How to cite this article: Sai Lakshmi I, Madhu Varma K, Girija S Sajjan, Kalyan Satish R, Sindhu D, Sowmya M. A comprehensive review on electrospinning design, parameters and potential use of electrospun nanofibers in regenerative endodontics.. Int J Dent Mater 2020;2(2): 37-44.
DOI: http://dx.doi.org/10.37983/IJDM.2020.2202

investigated by Zeleny in 1914 [1]. This electrostatic force stretches the Visco-elastic solution as it solidifies to fabricate electrospun fibres [2]. Electrospinning is simple in its basic set-up, thereby making this accessible to almost every laboratory. It is a versatile technology that is efficient in producing nanofibres suitable for various Bio-medical applications [3].

The technology of withdrawing ultra-thin fibres from visco-elastic fluid under a strong electric field was discovered about a century ago [4]. Electrodynamics led to the development of electrospinning to produce fibres, which was invented in 1902 by Cooley and Morton [5,6]. The term electrospinning was taken into records in 1993 by Darrell H. Renker [7,8]. Most of the time, materials for dental applications were found to be smaller in size and volume due to the size restriction of the oral cavity. The ability of electrospinning technology to produce fibres in sub-micron to nano-meter dimension and its flexibility in material selection led to the production of materials suitable for dental applications [2].

Electrospinning also aids in the incorporation of additives like medicaments to get desired properties in the final materials [9,10]. This technique has been widely employed for the fabrication of nanofibrous scaffolds with fibre diameter alignment tailorability and diversity in raw-material [11]. The tailorability of fibre diameter and pore size provides optimal conditions for differentiation and proliferation of cells [12]. Electrospun materials have the benefits of improved cellular interactions, enhanced protein absorption, which facilitates binding sites for cell receptors and high surface area to volume ratio [13].

2. Basic Set-up of electrospinning

Electrospinning set-up typically consists of four major components. which includes high voltage power supply, syringe with pump, metal tip needle (spinneret) and collector [14,15].

2.1 Principle

The basic principle of electrospinning involves potential voltage difference between the polymer solution flowing through a spinneret into the collector. When the potential difference overcomes the surface tension of the solution, then fluid jet splits to form fibres that are solidified with the evaporation of the solvent [16].

A detailed explanation of the conversion of visco-elastic polymer solution into nanofibres was shown in Figure 1.

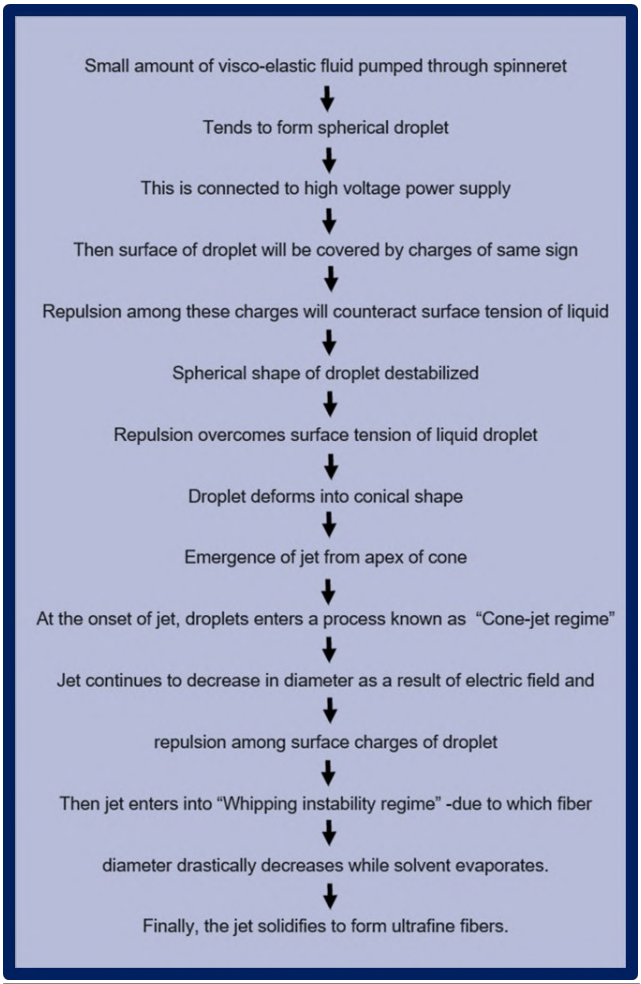

Figure 1. Steps involved in conversion of visco-elastic polymer solution into nanofibres.

2.2 Factors affecting fibre morphology and properties of electrospun fibres

The process of electrospinning produces continuous nanofibres through uniaxial stretching of visco-elastic solution [2]. To appreciate the process of nanofibre formation through electrospinning, different parameters that affect the process have to be considered. The key parameters affecting the properties of electrospun fibres and fibre morphology were described into four types. These parameters include processing, systemic, solution and physical parameters. The list of factors in each parameter affecting the characteristics of electrospun fibres was shown in Table 1.

2.2.1 Processing parameters

2.2.1.1 Voltage

Increase in voltage would discharge the polymer jet with a stronger repulsion, accelerates more volume of electrospinning solution, resulting in more stretching and decreased diameter of the fibres. However, an optimal voltage is necessary to initiate the polymer jet from the Taylor cone apex [17]. The applied voltage also

Table 1. Factors affecting the characteristics of electrospun fibres in each parameter.

Parameters	Individual factors
Processing parameters	Voltage
	Feed rate
	Distance of Collector
	Volumetric Flow rate
	Needle diameter
	Motion
Systemic parameters	Molecular weight
	Solvent
	Polymer type
Solution parameters	Viscosity
	Concentration
	Conductivity
	Surface Tension
	Dielectric constant
Physical parameters	Relative Humidity
	Temperature

had an effect on droplet shape prior to jet formation. Higher voltage results in an increased flow rate of solution and faster electrospinning [18].

2.2.1.2 Feed rate

The feed rate mainly determines the amount of available solution between the tip of the needle and electrospinning target. Increased feed rate causes the fusion of fibers due to improper evaporation of the solvent before reaching the collection point [18].

2.2.1.3 Distance of Collector

The reduction in the distance causes shorter flight time for the jet. So, it may not have sufficient time to solidify and results in the fusion of fibers. Increasing the distance drops the surface charge density, decreases the magnitude of the electric field, forming fewer charged ions [19]. This increase in distance results in elongation and decreases the diameter of the polymer jet.

2.2.1.4 Volumetric Flow Rate

Faster flow may stagnate the solution at the tip of the needle. As the rate of flow increases, the surface charge density decreases. The flow rate of the solution affects various features of nanofibres, such as diameter, porosity, and geometry [18]. A constant flow-rate is required to minimize the bead formation in electrospun materials [20]. Slow flow-rate reduces the diameter of the electrospun nanofibres [21]. In addition, the slow flow rate resulted in a smaller number of beads compared to a faster flow rate [22]. Therefore, in order to fabricate nanofibre continuously, the flow rate needs to be optimized [23].

2.2.1.5 Needle diameter

Fibre diameter was reported to increase with a greater needle tip diameter [24,25]. Smaller internal diameter reduces the clogging due to less exposure time of the jet to the environment. Reduction in inner needle diameter increases the solution surface tension corresponding to a smaller droplet that causes the jet to decrease its acceleration. So, jet gets more flight time before deposition and has more stretching and elongation; this results in smaller diameter fibres [23].

2.2.1.6 Motion

Regular electrospinning yields randomly aligned nanofibres [26]. Control on the geometry of the deposition of fibre or getting other desired fibre patterns can be achieved with a change in the design of the collector. One of them includes parallel bars with a gap in-between the two that leads to aligned nanofibres [27].

2.2.2 Systemic parameters

2.2.2.1 Molecular weight

Molecular weight represents the length of the polymer chain that, in turn, influences the entanglements. These will prevent the jet from premature splitting during the process. Higher molecular weight results in a viscous solution when compared to lower molecular weight [26]. Increasing molecular weight can result in decreased beading [28].

2.2.2.2 Solvent

The solubility and boiling point of the solvent are two essential factors to choose the desired solvent before electrospinning. Volatile solvents are considered to be an ideal option due to rapid evaporation and dehydration of the nanofibres [29]. A very low boiling point favours rapid evaporation, so this should be avoided to prevent the obstruction of needle orifice before electrospinning.

It was found that high boiling point solvents may not dehydrate completely before reaching the target resulting in a flat ribbon-shaped fibre instead of round fibre [30,31]. The volatility of the solvent might affect the features of electrospun nanofibres, including shape, porosity, and size. Hence, particular attention must be taken during the evaluation and selection of electrospinning solvents [30].

2.2.3 Solution-related Parameters

The solution properties are important to attain uniform fibres. It should have optimal low surface tension and high enough charge density and viscosity so that the collapse of the jet into droplets can be prevented before the solvent evaporates [32]. Polymer characteristics such as solution viscosity, concentration, surface tension, and solution conductivity influence the nanofibre morphology and properties.

2.2.3.1 Viscosity

Viscous solutions (optimum) enhance chain entanglements and result in uniform fibres without any beads. Less viscous polymer solution breaks up into small droplets or creates beaded fibres [33]. However, if the viscosity of the solution is too high, then it will be difficult to force the solution through capillary, and the solution at the tip may dry up [34].

2.2.3.2 Concentration

The concentration of the solution below the threshold value will result in the formation of droplets instead of fibres. The high concentration of the solution increases viscosity and may lead to processing problems. Increasing concentration can result in decreased beading and increased fiber diameter [35].

2.2.3.3 Effect of Conductivity

High conductivity facilitates polymer solution to carry greater charge compared to low conductivity. Hence, high conductivity yields greater tensile forces to applied voltage and reduction in nanofibre diameter [36]. Fong *et al.* examined the effect of sodium chloride on polymer for the fabrication of electrospun nanofibre and reported a higher charge density of electrospinning jet. This increased charge density results in the formation of smooth and uniform nanofibre [33]. Increasing conductivity through the addition of salt can result in defect-free, smaller diameter fibers [33,37].

2.2.3.4 Surface tension

Surface tension results in decreased surface area of the solution and aids in the formation of a spherical droplet. In case of low concentration, a high ratio of solvent molecules have an increased tendency to assemble and form a spherical or bead formation [33]. Low surface tension solvents should be used to get bead free uniform fibres.

2.2.3.5 Solvent dielectric constant

Increasing dielectric constant can result in decreased bead formation. The solvent with a higher dielectric constant has a higher density in solution [38].

2.2.4 Physical parameters

2.2.4.1 Relative humidity

Humidity causes changes in the diameter of the nanofibres by controlling the solidification process of the charged jet. An increase in humidity results in a decreased diameter of nanofibres. Further, an increase in humidity led to bead fibre for individual polymers and almost no electrospinning for the blends [39].

2.2.4.2 Temperature

Temperature causes two opposing effects to change the average diameter of the nanofibres: (i) it increases the rate of evaporation of the solvent and (ii) it decreases the viscosity of the solution. The increase in the evaporation of the solvent and the decrease in the viscosity of the solution work by two apposite mechanisms, however, both lead to a decrease in the mean fibre diameter [40].

3. Applications of electrospun nanofibres in regenerative endodontics

Pulp-dentin complex regeneration aids in extending the normal function of the natural dentition, especially in cases of traumatized permanent immature teeth, which hinders completion of root development and maturation [41,42]. The idea behind the regenerative endodontics is mainly based on stem cells' capacity to regenerate. These stem cells will be introduced into root canals through the intentional laceration of periapical tissue after a thorough disinfection protocol. The growth factors and stem cells from the apical area populate the scaffold, inducing tissue regeneration [43,44]. Therefore, both root canal disinfection and blood-clot formation have been shown to play a critical role in new tissue formation and overall root maturation and development.

The application of the antibiotic mixture in regenerative endodontic procedures was introduced in 2001 [45]. Since its emergence, this intra-canal antibiotic paste, i.e., either Triple antibiotic paste (TAP) or Double antibiotic paste (DAP), has been the most commonly used inter-appointment medicament [46]. Even though root canal irrigation with sodium hypochlorite (NaOCl) associated with antibiotic mixtures (i.e., TAP or DAP) has led to maximum bacterial elimination, but their use at high concentrations have been shown to negatively

impact dental derived stem cells survival and function [41,47]. It was shown that the widely used creamy paste (1 g/mL) of the triple antibiotic mixture is toxic to stem cells from the apical papilla (SCAPs) [48].

It was stated that Triple Antibiotic paste concentrations ranging from 0.01 to 0.1 mg/mL were not cytotoxic when applied directly onto the stem cells from apical papilla (SCAP) [48] and had no effect on viability after its removal from the root canal lumen [49]. However, antibiotics mixed with water or saline in such low concentrations result in a watery mixture that cannot be retained inside root canals. Therefore, it would be beneficial to use a biocompatible nanofibre-based intracanal drug delivery construct to release antibiotics at lower yet anti-microbially effective concentrations [50].

In drug delivery systems, coating the electrospun fiber with a shell is considered to be effective in controlling the release kinetics of the drugs [51]. The shell coat serves as an outer protective layer. Hydrophilic drugs can be incorporated in the core phase and hydrophobic polymers in the shell phase. In core-sheath nanofibers, the core swells or dissolves, forming pores in the shell after the dissolution of hydrophilic portion in the core, thereby allowing for the sustained release of the drug [52].

In order to overcome the inherent toxicity of the agents mentioned above, the concept of a cell-friendly disinfection strategy was introduced through the successful development of novel antibiotic-containing polymer nanofibres [43,50,53]. These novel drug delivery systems designed for regenerative endodontics are predicated on the fact that controlling the antibiotic dose and release rate will lead to enhanced stem cell viability while preserving anti-microbial activity [43,50,54-57]. In electrospinning, a polymer solution containing the desired concentration of antibiotics is prepared to produce nanofibres [43,50].

The reason behind the use of antibiotic-containing nanofibres as a three-dimensional (3D) tubular drug delivery construct [53,58] is based on the fact that the addition of low antibiotic concentrations and the slow drug release provided by these nanofibrous constructs will be able to eradicate the infection and thus create a bacteria-free environment favourable to tissue regeneration [56,57,58]. Importantly, in this strategy, the anti-microbial agents are delivered directly onto the dentinal walls, where microbial biofilms have been found to be present.

Collagen or Polycaprolactone (PCL) gelatin-based nanofibrous scaffolds incorporating bioactive glass nano particles were developed for dentin-pulp regeneration and showed enhanced growth and odontogenic differentiation from human dental pulp stem cells (DPSCs) compared to collagen nanofibrous scaffold via the integrin-mediated process [58,60]. Bottino *et al.* incorporated antibiotics (metronidazole and ciprofloxacin) to polydioxanone (PDS) electrospun scaffolds and observed that these scaffolds were more effective at delivering antibiotics. They require a lower dose against pathogenic bacteria, including Porphyromonas gingivalis and Enterococcus faecalis, compared to drugs delivered via pastes [50].

It was shown that these electrospun meshes of PCL have a strong potential for promoting odontogenic growth and differentiation, as suggested by increased turnover of collagen I and other proteins when tested in vitro with human pulpal cells [61]. Taken together, the major advantage of electrospinning might be its ability to produce complex geometry of nanofibrous scaffolds for dentin-pulp complex regeneration.

Electrospinning was used to fabricate tubular 3D drug delivery constructs comprising polydioxanone and three antibiotics (metronidazole, ciprofloxacin, and minocycline) at a much lower concentration than in the triple antibiotic paste. The 3D construct was designed to smoothly fit within the individual anatomy of immature teeth, that is, a parallel and tubular thin root dentin wall. The tubular 3D triple antibiotic-eluting drug delivery constructs were found to be effective in ablating intracanal biofilm in a similar fashion to the well-established triple antibiotic paste [62].

In electrospinning, the chosen polymer solution can be incorporated with one or a combination of antibiotics, making it possible to fabricate fibres with a narrow or wide spectrum of action (e.g., ciprofloxacin (CIP), metronidazole (MET), and minocycline (MINO) that have been shown to inhibit the growth of endodontic pathogens [50,56,57]. In a study, ciprofloxacin-containing polymer nanofibres were tested against E. faecalis biofilm developed on human root fragments and found maximum bacterial biofilm elimination [55].

4. Conclusion

Nanofibres used for the seeding of regenerative cells (like dental pulp cells, mesenchymal cells, odontoblasts, growth factors, etc.) provide a porous 3D surface that promotes regeneration of teeth [50]. Apart from this, the nanofibres produced by electrospinning offers various advantages like adhesion to cells, mimicking extracellular matrix, differentiation, and proliferation

of cells. In fact, scaffolds containing antibiotics have been proven to reduce or completely eradicate infection by the controlled release of a wide variety of antibiotics [63]. Ultimately, the nanofibrous scaffold's ability to deliver intracanal, controlled amounts of antibiotics might have positive treatment outcomes conducive to tissue regeneration by rendering a bacteria-free environment while minimizing the toxic effects associated with the use of the antibiotic paste.

Conflicts of interest: Authors declared no conflicts of interest.

Financial support: None

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Review Article International Journal of Dental Materials 2020; 2(2) ISSN: 2582-2209

Exploring Best Fit-Dental Materials for CAD/CAM

Dr. Payal*

Tutor, NIMS Dental College and Hospital, UDMRI Dehradun, India

INFORMATION

Article History

Received 12th May *2020*
Received revised
14th May 2020
Accepted *18th May 2020*
Available online
31st May 2020

KEYWORDS

CAD/CAM Dentistry

Ceramics

Zirconia

Resin

PMMA

Nanoceramics

ABSTRACT

With the extensive research and investment in CAD/CAM technology, the available options of machinery, as well as dental materials, are growing day by day. To find the best fit, one must have a thorough understanding of what material and process are apt for a particular situation, and one should also keep oneself updated with the latest findings and products provided by various manufacturers. With the new technology coming in, cost becomes a significant factor in deciding the right material. There are plenty of options, but the question is 'are these options scalable in terms of manufacturing at scale and low cost?'. We would try to explore the options available in the market and the ones that are in the pipeline in the document. Three factors affecting the decision are strength, aesthetic quality and cost. Necessity is to find that fine balance among these three as per the clinical situation.

1. Introduction

CAD/CAM Dentistry refers to utilising the intelligence and precision of a computer in designing and manufacturing a customised patient-specific dental fixture or device [1]. While Dentistry is as ancient as the human civilisation and CAD/CAM has its roots to ancient Egypt, Greece and Rome when Leonardo Di Vinci had used modern graphics convention in his works, the amalgamation of both happened in the 1970s. The utilisation of the CAD/CAM technology in dentistry marks its start in 1971 with Dr Duret as he used optical means to take an impression of abutment teeth [2]. He then forged a crown with the help of that impression using a numerically controlled machine. A commercially designed CAD/CAM system was first introduced in 1985 by Mormann, that was named as CEREC [3]. Currently, CEREC is widely used across the globe for the fabrication of inlays, outlays, crowns and many other dental fixtures and devices. Modern CAD/CAM provides an alternate and efficient method to process fixed dental prosthesis and indirect dental restoration. It eliminates many laboratory and clinical steps involved to make the process faster and efficient. It promises esthetic and accurate restorations in quick time.

The process is mainly comprised of two things, firstly, the machining system that facilitates the scheme, secondly, the milled materials that essentially define the long-term success or failure of the system. The milling material must be damage resistant, to be polished, glazed or stained easily and most importantly milled rapidly. Enhanced systems may also be used to mill high strength ceramic materials. A few systems are capable of milling materials like Titanium or noble or base material. As a final step, the milled framework needs to be veneered using porcelain powder by hand or pressed with prefabricated ingots [4].

Everyday advancing digital fabrication techniques and development of stronger and with enhanced characteristics, ceramic materials have provided practitioners

Correspondence: *Corresponding author Email Address: payalsingh2175@gmail.com

How to cite this article: Payal. Exploring Best Fit-Dental Materials for CAD/CAM. Int J Dent Mater 2020;2(2): 45 -51. *DOI:* http://dx.doi.org/10.37983/IJDM.2020.2203

with multiple options and ability to combine durability with aesthetics. But with increasing options, it becomes difficult to determine the best fit for a particular situation. Different CAD/CAM materials have different material properties, clinical indications and processing techniques. All of them together defines the best use of a particular material. Thus, the practitioner must understand the properties and required processing techniques of CAD/CAM materials available. [5].

2. Different classes of materials

The first inlay fabricated by chairside CAD/CAM was made with a ceramic block made of fine grain feldspathic ceramic in 1985. Different systems have been developed since then with the help of a series of development in software and hardware. Modern systems offer 3D design programs combined with improved machinery that can produce frameworks as complicated as custom lithium disilicate implant abutments. CEREC I was the first system that used feldspathic ceramics for smaller occlusal inlays. Reinforced ceramics were developed to extend the indications of restorations. Pre-crystallised stage of such reinforced ceramics is used to enable rapid milling. Crystallisation needs to be done post milling to get the mechanical strength and final colour. Next set of materials used are of resin class that is soft and less susceptible to brittle fracture. These materials are then mixed with ceramic particles to improve their mechanical properties. A ceramic network is infiltrated into resin polymers in the process to combine advantages of both materials. A few metals have also been used as a block, but these instances are rare.

2.1 Metals and alloys

Titanium and Cobalt-Chromium (Co-Cr) alloys are the most common metals used in milling Fixed Partial Dentures (FPDs). Pre-sintered form of Co-Cr alloys is used that enables faster machining and low damage to burs. Co-Cr blocks are made by compaction of Co-Cr powder by isostatic pressure. It gives soft and tender but solid metal block. Sintering the prosthetic fixture as a post-treatment improves the mechanical properties. This treatment reduces the pores and improves its properties regarding compactness. Precious metal alloys couldn't become popular due to various reasons, including high cost.

2.1.1 Usages

Lack of aesthetics is the major roadblock for widespread usage of the material. Though it is not used for achieving abutments but is widely used in thin reconstitution. Titanium is preferred for metal implants as electrochemical corrosion is the least for Titanium. It becomes more important in cases where metal restoration has already been done. These blocks are suitable for frameworks and metal-ceramic restorations. However, metal blocks are not used in chairside CAD/CAM. A few popular blocks present in the market are Sintron produced by Amann Girrbach and Crypton produced by Dentsply [6]. The primary difference between these two is the milling process; former requires dry milling whereas later is milled with oil and water spray in a closed environment as suspended Co-Cr microparticles are toxic.

2.1.2 Implementation

Metal blocks are sintered post-producing the prosthetic framework as it requires a post milling treatment. It is basically a heat treatment process that is performed with or without external pressure depending upon the required material characteristics where a porous material reduces its porosity resulting in better compactness. Sintering causes around 10 per cent shrinking of the parts; thus, the design of the part must incorporate this to achieve exact size after the treatment.

2.2 Ceramics

Metals are strong, but they don't provide a natural appearance. The demand for natural appearance resulted in the development of ceramic restorative material. Ceramic is a composite of two or more substances in the form of glass or polycrystalline matrix. Fillers in different quantities are infused to obtain certain mechanical properties, and particles (glassy or crystal) or modified atoms are doped to obtain stabilised polycrystalline structure. The glassy matrix defines the aesthetic property, greater the glass rate; more would be the translucency (diffusion of light) to best imitate the properties of enamel and dentin [7]. There is always a tradeoff between aesthetics (translucency) and strength in dental ceramics. Crystalline ceramics provide higher strength but have an opaque appearance. Such ceramics with feldspathic porcelain veneer are generally used in the bilayer restoration framework. On the other hand, predominantly glassy ceramics provide high translucency but are relatively weaker than crystalline ceramics. Based on microstructure, strength, aesthetic quality and clinical indications, ceramics are divided into three major classes such as Zirconia, Crystalline glass ceramics and Resin ceramic composites.

2.2.1 Zirconia

It is the most popular among the CAD/CAM materials used in dentistry. It was introduced around 2005, and it quickly gained popularity as it has excellent mechanical properties and is easily machinable in CAD/CAM machines. It is basically an oxide with high tensile strength, hardness and corrosion resistance. Most of the material used is obtained from Zirconate (ZrO_2-SiO_2, $ZrSiO_4$) and Baddeleyite (ZrO_2). The basic difference between them is the amount of Zirconia present in them. While Baddelyite contains 96.5 to 98.5 per cent [8], Zirconate requires significant processing to get Zirconia. But Zirconate is significantly abundant than Baddelyite. Zirconia (ZrO_2) presents a monoclinical structure at room temperature. It is processed at high temperature synthetically to form cubic zirconia (a cubical structure). Cubic Zirconia is harder, translucent and optically flawless.

2.2.1.1 Phases of Zirconia

Zirconia exhibit polymorphism, and it has three crystal structure or phases characterised by distinct crystallographic structures: monoclinic, tetragonal and cubic (Figure 1). Monoclinic structure characterises pure zirconia that is stable up to 1170°C. Above 1170°C tetragonal zirconia is formed, and after reaching a temperature of 2370°C, cubic zirconia starts forming. Tetragonal phase changes to monoclinic at 970°C after processing [8]. Cooling of zirconia results in a significant volume change of around 2-3% due to polymorphism. It can't be processed at high temperature as the change in volume is sufficient to exceed the fracture limit and develop cracks and fatigue in the framework. Thus, manufacturing of components from pure zirconia is complicated, stabilising oxides are added that helps in maintaining the tetragonal structure at room temperature. The transformation of the crystal structure is utilised to develop particular mechanical property of zirconia such as tenacity. It transforms from tetragonal to monoclinic between 850°C and 1000°C depending upon the magnitude of strain energy.

One of the most commonly used zirconia is Yttria. It is a stabilised form in which tetragonal phase of Zirconia is stabilised at room temperature by adding Yttria. Another main additive for zirconia is Alumina, the amount of Yttria and Alumina decides the strength and translucency.

2.2.1.2 Types of Zirconia

2.2.1.2.1 Framework Zirconia

It is used in multi-unit bridge framework for posterior and anterior regions. It is an aesthetic alternative to Metal restorations and is being widely adopted.

Properties and indication

It is diffused with feldspathic porcelain or glass-ceramic to provide a natural appearance. It has higher alumina content around 0.25% that makes it strong and opaque. The usual composition of framework Zirconia is 3-mole percentage Yttria stabilised tetragonal Zirconia [10]. Transformation toughening provides zirconia with excellent mechanical properties. The framework zirconia expands on the application of stress which inhibits crack propagation. It has a flexural strength of 900 to 1400Mpa and fracture toughness of 5 to 9 MPa/m2 [5].

2.2.1.2.2 Full Contour Zirconia

It is the most popular choice for moral single crowns and posterior multi-unit bridges as an alternative to FPM restorations and gold crowns.

Properties and indications

It has a low content of alumina around (0.05%) [5]. Thus, it has improved translucency that makes it more suitable as a single layer or monolithic material. Monolithic restorations are done in a single process, therefore, better suited for posterior region. Full-contour Zirconia also has 3-mole percentage Yttria stabilised zirconia. Thus, it also has fracture toughness and flexural strength like Framework Zirconia. While using monolithic Zirconia, wear of antagonist enamel was always a roadblock; this has been solved by polished zirconia that maintains a polished surface finish.

2.2.1.2.3 Cubic Zirconia

It is the most popular choice for single crowns and anterior three-unit bridges. It is a tougher alternative to glass ceramics and a more aesthetic alternative to full contour Zirconia.

Properties and indications

It has more than 5 moles per cent of Yttria and a higher proportion of cubic phase of Zirconia. This gives it translucency and makes it look more natural. Though, the aesthetic quality of cubic Zirconia is better than Full Contour Zirconia but not as good as feldspathic porcelains or glass-ceramics. The higher proportion of cubic phase also causes brittleness resulting in less resistance to crack propagation. It has a flexural strength of 500 to 700 MPa, and its fracture toughness has not been measured accurately.

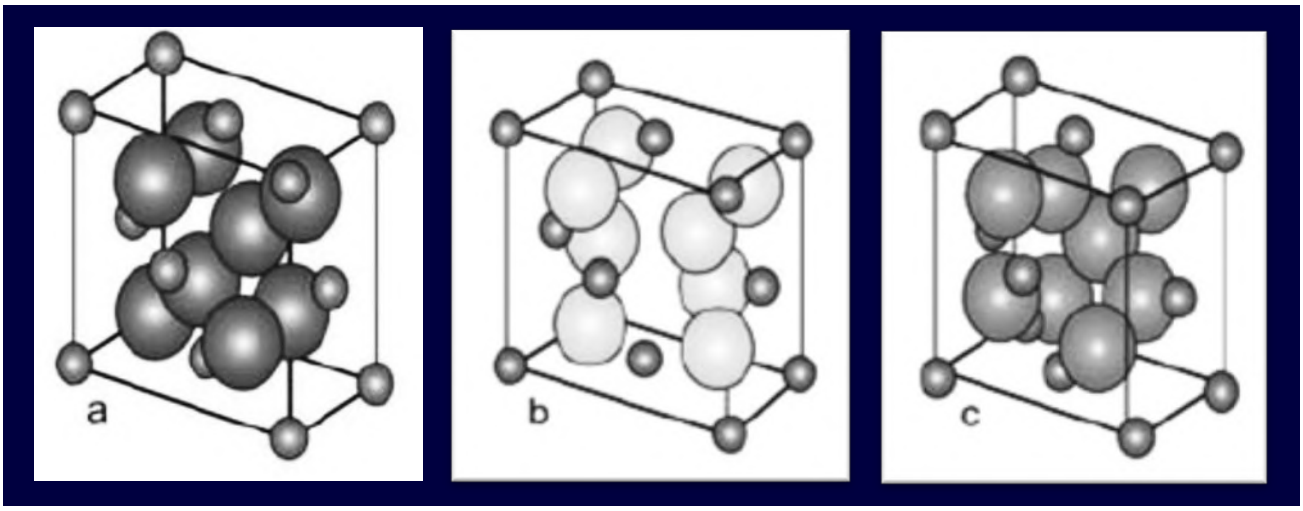

Figure: 1. Zirconia crystalline structures; a. Monoclinic b. Tetragonal, and c. cubic crystal structure [9]

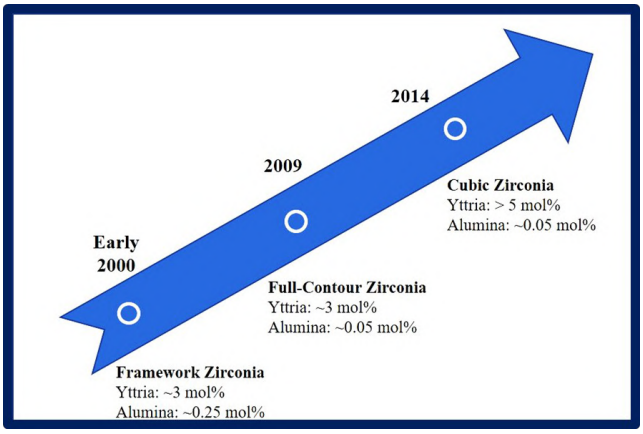

Figure 2. Evolution of Zirconia types , Time-line and Constituents [10]

The development of various types of zirconia materials are described in figure 2.

2.2.1.3 Application of Zirconia in Dentistry

There are various zirconia ceramic systems available. Yttrium cation-doped tetragonal zirconia polycrystals (3Y-TZP), magnesium cation-doped partially stabilised zirconia (Mg-PSZ) and zirconia- toughened alumina (ZTA) are the ones used till now in dentistry [10]. These materials have been used in dental posts (Figure 3.a), crown and bridges (Figure 3.b), implant abutments (Figure 3.c), and aesthetic orthodontic brackets [11].

2.2.2 Crystalline glass ceramics

Glass Ceramics have become popular because of its superb aesthetic quality and ability to bind with tooth structure. It is mostly used in the anterior regions as it does not have sufficient strength to be used in the posterior regions. It is a multiphase solid that contains finely dispersed crystalline phase with a residual glass

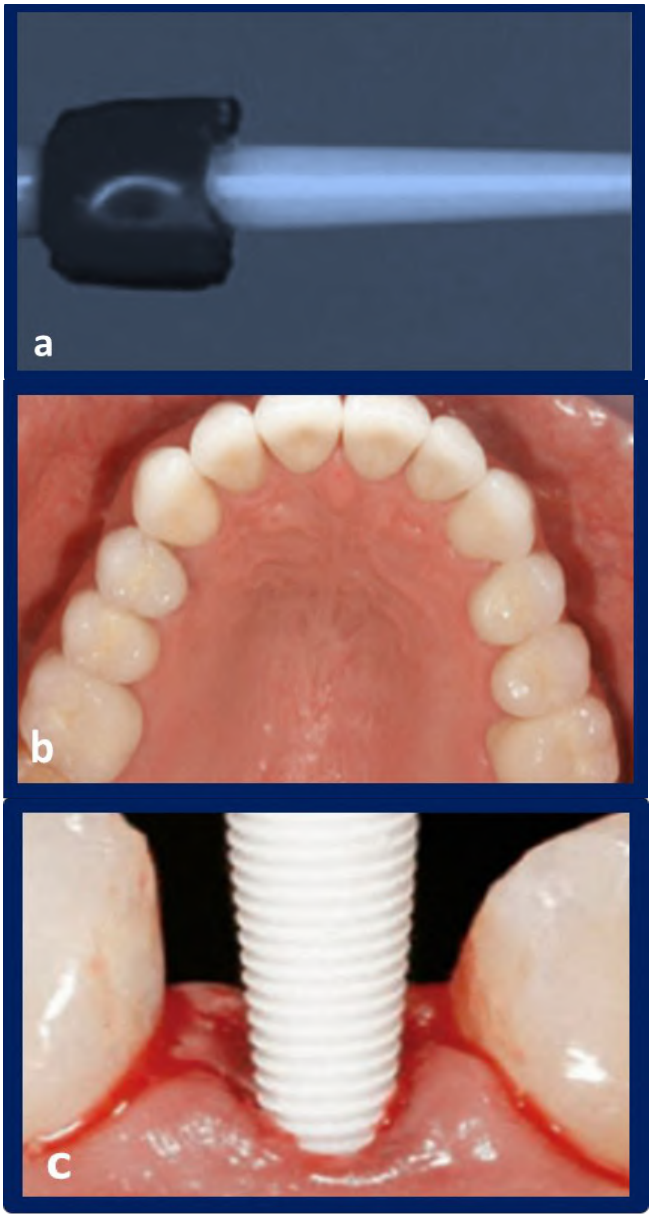

Figure 3. Zirconia-based a. Dental posts , b. Frameworks, and c. Implant [12].

phase. Controlled crystallisation of the glass is done using creaming heat treatment method to form tiny crystals evenly distributed throughout. The size of the crystals, their number and growth are controlled via temperature and time of the heat treatment process [12].

The first CAD/CAM ceramic material used by dentists was feldspathic porcelain that was followed by leucite -reinforced with better flexural strength. These became popular because of their excellent aesthetic properties and were mainly indicated for inlays and veneers. Now, it is being replaced with stronger glass-ceramics. Similar to feldspathic porcelain, glass ceramics has a glassy matrix structure that provides translucency and a crystal phase embedded in the structure that imparts mechanical properties.

2.2.3 Lithium disilicate ceramics (LDS)

LDS is available in the market in many varieties with the excellent combination of translucency and strength. Anterior single crowns and three-unit anterior bridges are made with less translucency and high strength blocks whereas, inlays, onlays and veneers are made with high translucency and low strength blocks. LDS is also an option for higher aesthetic demand and somewhat for single posterior crowns.

Properties and indications

LDS has a flexural strength of 200 to 400MPa and fracture toughness of around 2 to 2.5 MPa/m2 [13]. Initially, it was a sub-structural material that needed to be veneered with feldspathic porcelain. With certain improvements in aesthetic and mechanical properties, it became usable as a single monolithic layer. LDS has better aesthetic quality than all the Zirconia based materials and more strength than other glass-ceramics. LDS is comparatively more abrasive, so dentists polish the restoration to remove milling marks before going for sintering.

2.2.4 Leucite-based Glass Ceramics

These are formed by controlled nucleation and crystallisation of the base glass. The base glass is composed of additives that facilitate nucleation and crystallisation process in K_2O-Al_2O_3-SiO_2 system. The final step is heat treatment after which leucite crystals are precipitated containing 35 to 45 % crystal content by volume and crystal size of 1 to 5µm [13].

Properties and indications

Leucite-based ($K[AlSi_2O_6]$) glass-ceramics exhibit superior biocompatibility alongside their suitable physical, chemical and mechanical properties. They are also suitable for chair-side milling process. Leucite based products are available in different colours with different level of translucency and brightness that enables better imitation of natural teeth. Due to its excellent aesthetic quality, it is used for fabricating anterior crowns, inlays and onlays. It is also a handy material as the complete process of pre-operative condition to the fabrication of the restoration to cementation of restoration takes around 2 hours.

2.2.5 Yttrium-stabilised zirconium oxide-based ceramic

Zirconium oxide-based ceramics are mainly used in fabricating crown and bridge frameworks. It is also suitable for post, abutment and implant. Fabrication of dental restorations with Zirconium oxide is achieved through machining of dense ceramics and pre-sintered ceramics. While machining of dense ceramic is time-consuming and the required machinery and equipment are heavy, another method is developed where it is milled in a porous state using smaller and less complicated machinery.

Properties and indications

These are characterised by high fracture strength and toughness. Flexural strength measures around 900 to 1200 MPa and fracture toughness around 4 to 5 MPa/m2 [13]. Restoration achieved from CAD/CAM equipment is densely sintered at higher temperatures between 1400 to 1500°C. Colouring of Zirconia based ceramics have also been made possible recently making it aesthetically more viable [13].

2.3 Resins

Resins are used as restorative as well as adhesive. Its micro retention property makes it more suitable for filling smaller cavities where other restorative materials don't hold well [14]. Conventional resins are made up of polymeric matrix reinforced by inorganic fillers like glass or glass-ceramic or oxide ceramic [15]. Currently, there are mainly three variations of resin materials available. They include Polymethyl Methacrylate (PMMA), Resin composite, and Nanoceramics

2.3.1 Polymethyl Methacrylate (PMMA)

These are made of methyl methacrylate polymers. There are no added fillers, thus resulting in lower mechanical strength. It is a thermoplastic polymer that is transparent.

Properties

It has a tender structure that enables fast and easy machining and causes minimum wear to the bur. It is used for making temporary restorations or any prosthesis that is to be used for a maximum of 1 year.

2.3.2 Resin Composites

These are matrix resins composed of monomers infused with inorganic fillers. Fillers are to improve the mechanical properties of the composite. More the quantity of the charge better is the mechanical property of the resin. Smaller load size results in improved surface finish, wear-resistance and aesthetics[16]. The composite blocks are made by thermal polymerisation under a pressure of several thousand bars. The conversion rate of these blocks in chairside machining is more than 90% as compared to 50 to 60% for other blocks [17].

Properties and indications

This is the only resin used in chairside CAD/CAM. Its wearing period is three years as compared to 1 year for PMMA. The machining for resin composites is easier and causes less wear to burs. The flexural strength of Resin composite is around 80 MPa that makes it viable for making the veneers, inlays and onlays, anterior bridges, posterior bridges, anterior crowns and posterior crowns.

2.3.3 Nanoceramics

It has similar microstructure to resin composites. It has a polymeric matrix and has ceramic nanoparticles as fillers. Fillers have sizes lesser than 100nm and constitute 80% of the weight. The fillers can be conventional ceramic, zirconia or a mixture of both.

Properties and indications

The characteristics of Nanoceramic are quite similar to the natural tooth. It has a flexural strength of 200MPa, compression of 380MPa, the elasticity of around 15GPa and abrasion of approximately 2 to 10 microns/year [6]. Nanoceramics are used for veneers, single anterior crowns, single posterior crowns, inlays/onlays, anterior bridges and posterior bridges. These are easily machinable due to its softer matrix structure, and it doesn't require any milling treatment other than make-up photopolymerisation.

2.4 Polymer Infiltrated Ceramic Network Material (PICN)

It has a hybrid structure with a matrix of polymer and ceramic penetrating into each other. It has properties of ceramic as well as polymer. The fabrication process requires two steps, first being the production of a pre-sintered ceramic network that is porous and then conditioned with a coupling agent. Secondly, it is penetrated with a polymer [6].

Properties and indications

It has characteristics similar to dentin as its abrasion, elasticity, and flexural strength is much similar to dentin. It is more wear-resistant than resin composite due to the ceramic network present in the structure. This material is quite recent; thus, the exact characteristics are not defined. However, it has looked promising, and research has been going on for further improvements. Currently, it is used for fabricating veneers, inlays/ onlays, single anterior crowns, posterior single crowns and implant prosthesis [6].

3. Conclusion

The new generation of CAD/CAM materials offers a plethora of options in terms of fabrication techniques as well as varying properties. While zirconia has become most popular due to its low cost and viable mechanical and aesthetic properties, new materials produced by infusion techniques promises enhanced properties in terms of aesthetics as well as strength. Glass based composites are aesthetically superior and they are being imparted with better mechanical properties using additives and different treatment techniques. Materials like nanoceramics and PICN promises better quality in terms of both strength and natural look. For inlays, onlays and veneers, adhesive cementation and natural look are critical factors as most of the materials fulfil the strength requirement. LDS and resin ceramic composites are best suited for them as they provide good aesthetic quality with greater machinability, thus reducing the chair time.

Similarly, for anterior crowns, LDS provides the most suitable combination of mechanical and aesthetic properties. Different materials are best suited for various clinical situations. However, none of these has properties for universal application. The passionate effort is being put in the research to enhance strength, aesthetics, machinability, ability to bond with other dental substrate and enhanced durability of these materials.

Conflicts of interest: Authors declared no conflicts of interest.

Financial support: None

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Review Article International Journal of Dental Materials 2020; 2(2) ISSN: 2582-2209

Recent advances in metallurgy and design of rotary endodontic instruments: a review

Aparna Palekar [1], Akhilesh Vajpayee[2,*], Basawaraj Biradar[1]

[1]Professor, [2]Postgraduate Student, Department of Conservative Dentistry and Endodontics, Rural Dental College, Pravara Institute of Medical Sciences, Loni, Maharashtra, India.

INFORMATION

Article History

Received *10th May 2020*
Received revised
17th May 2020
Accepted *18th May 2020*
Available online
31st May 2020

KEYWORDS

Nickel-Titanium

Heat-Treatment

M-Wire

R-Phase

Control Memory

ABSTRACT

A variety of instruments are available for the extirpation of the pulp and the instrumentation and preparation of the root canal. Recently, nickel-titanium (NiTi) alloy is utilised for the manufacturing of endodontic instruments. Compared to other metals, these alloys are highly flexible, which significantly enhances ease of canal shaping. This review article gives an account in the advances of NiTi endodontic instruments with an emphasis on metallurgical, mechanical properties, the design features of each generation with a special focus on the latest generations of NiTi instruments.

1. Introduction

To overcome some of the undesirable characteristics of stainless-steel files, a new generation of endodontic instruments made from nickel-titanium has added a new dimension to the practice of endodontics. The NiTi alloys started at the beginning of the '60s by W. H. Buehler, a metallurgist investigating nonmagnetic, salt resistant and waterproof alloys for the space program at the "Naval Ordinance Laboratory", in Silver Springs, Maryland, USA. This was named "NITINOL based the elements from which the alloy was composed; Ni for Nickel, Ti for titanium and NOL for the Naval Ordinance Laboratory. Nitinol is the names given to a family of intermetallic alloys of Ni and Ti, which possesses unique properties such as shape memory and superelasticity [1]. The first investigation of nickel-titanium in endodontics was reported in 1988 by Walia, Brantley and Gerstein using #15 files fabricated from nickel-titanium orthodontic alloy.

The unique property of super-elasticity allows to carry out extraordinary conservative shapes, and it can also be better centered. Furthermore, it provides less canal transportation and in this manner with more regard of the original anatomy. Advanced instrument designs have been developed to reduce the preparation time, improve working safety, and create a conical flare of preparations and continuously tapered shapes. The advanced instrument designs include with non-cutting tips, radial lands, different cross-sections, superior resistance to torsional fracture and with varying tapers [2].

Correspondence: *Corresponding author Email Address: *akhilv91@gmail.com*

How to cite this article: Palekar A, Vajpayee A, Biradar B. Recent advances in metallurgy and design of rotary endodontic instruments: a review. Int J Dent Mater 2020;2(2): 52-59.
DOI: http://dx.doi.org/10.37983/IJDM.2020.2204

2. Temperature Induced Phase Transformation

The crystal structure of NiTi alloy at high-temperature range (10000C) is stable with body centered cubic lattice (BCC), which is referred to as the austenite phase or parent phase. Nitinol shows dramatic changes in its yield strength, modulus of elasticity (MOE), and elastic resistivity on cooling through a critical transformation temperature range (TTR). Also, changes the crystalline structure occurs on reducing the temperature through this range. This change in crystalline structure is known as martensitic transformation. This phenomenon causes a change in the physical properties of the alloy and gives rise to shape memory characteristic (Figure 3) [3].

The atoms in this file are rearranged into a closely packed hexagonal array on placing into a curved canal. This atomic rearrangement imparts a more flexible character with a martensite crystal structure to the alloy. The molecular transition enables these files to bend easily around severe curves without permanent deformation. The alloy reverts to its original austenite form on the removal of the stress, and this is called as stress-induced martensitic transformation, which is a unique property of NiTi alloy and thus makes this material suitable for use in rotary endodontic instruments [4].

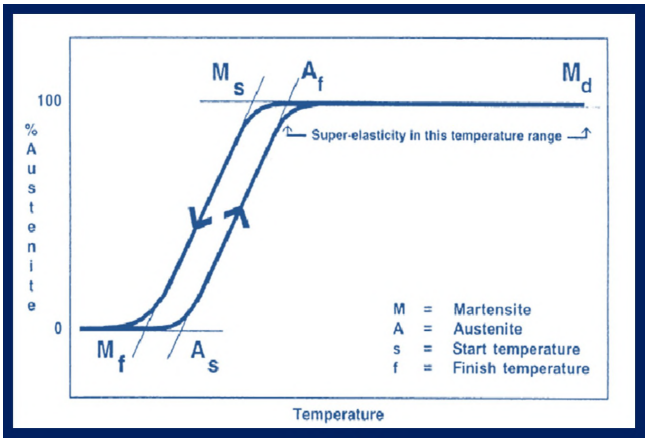

Figure 1. Temperature dependent transitions from austenite to martensite; where A_s: The starting temperature for transformation to austenite, A_f: The temperature at which transformation to austenite is finished. M_s: The starting temperature for transformation to martensite. M_f: The temperature at which transformation to martensite is finished.

3. Strategies in the alteration of NiTi alloy

In recent years, new forms of NiTi are developed by modifying the alloy by variations in metal processing and file manufacturing or correcting the surface defects.

3.1 Plasma immersion ion implantation

Conrad *et al.* and Tendys *et al.* introduced Plasma immersion ion implantation (PIII) in the late 1980s. The specimen is introduced in a chamber and immersed in the plasma, and a highly negative pulsating voltage is then applied to the sample. Briefly, it is a line-of-sight process in which ions are extracted from plasma, accelerated, and bombarded into a device. Ionic implantation brought differences in surface characteristics, an increase in the cutting efficiency, and improved wear resistance as shown by Rapisarda *et al.* [5].

3.2 Oxide formation on NiTi/Titanium oxide coating

Titanium has a higher affinity with oxygen compared to Nickel. So, with increased exposure time at moderate temperature, the oxide formation is composed mainly of TiO_2 with slow formation and growth. A study on the mechanical behaviour of the endodontic instruments and its corrosion resistance in Sodium Hypochlorite (NaOCl) solution was carried out by Aun DP *et al.* [6]. They observed an improvement in cutting efficiency and high resistance to corrosion on immersing in NaOCl solution. The coated instruments showed better performance in fatigue life after corrosion, so they concluded that this characteristic should be maintained since the TiO_2 layer can support relatively large deformations. Coating the endodontic instruments with a flexible TiO_2 protective layer with the help of dip-coating sol-gel method improves cutting efficiency, corrosion behaviour and resistance to fatigue failure.

3.3 Thermal nitridation

Powder Immersion Reaction Assisted Coating (PIRAC) is a nitriding method producing TiN on NiTi. Such modified surface consists of an outer layer of TiN which is thin and a thicker Ti2Ni layer underneath [7]. By placing a TiN layer on commercial rotary NiTi instruments corrosion resistance of files placed in contact with 5.25% NaOCl significantly increases.

3.4 Cryogenic treatment

Various metals are treated with deep dry cryogenic methods to enhance resistance to corrosion and wear.

These methods also improve strength and microhardness of metals. The entire cross-section of the instrument is affected rather than no change in the elemental crystalline composition on the surface of the alloy [8]. There are two mechanisms involved. In the first mechanism, the crystalline transformation from austenitic to complete martensitic following CT occurs. In the second mechanism, finer carbide particles precipitate within the crystalline structure (Figure 2). Kim *et al.* [9] observed that cryogenically treated instruments had significantly higher microhardness.

3.5 Electropolishing / Reverse plating

Electropolishing (EP) is a typical surface treatment process for final finish during the manufacturing of NiTi instruments. This process includes alteration of the surface chemistry and morphology takes place as surface imperfections are removed as dissolved metal ions. Once, the instrument is immersed in a temperature -controlled bath of electrolyte it serves as the anode when connected to the positive terminal of a direct current power supply, and the negative terminal is attached to the cathode. The surface of a metal oxidises as the current passes and dissolves in the electrolyte. A reduction reaction is observed at the cathode that normally produces hydrogen. Most often the electrolytes used are mixtures of concentrated solutions of sulfuric/phosphoric acid with a high viscosity. Bare NiTi surfaces are produced from Ti oxides with Ni concentrations from 2% to 7% depending on the electrolytes and regimes employed. In the process, the metal exhibits better corrosion resistance along with improved surface characteristics. Anderson *et al*. reported that the instruments with electropolishing show better resistance to cyclic fatigue loads and poor resistance to static torsional loading. The benefits of electropolishing are likely caused by a reduction in surface irregularities that serve as points for stress concentration and crack initiation [10]. Lopes *et al.* found significant increases in cyclic fatigue resistance and exhibiting fine surface cracks which assumed an irregular or zigzag path, that EP instruments demonstrated. In contrast, the non-EP files had cracks running along the machining grooves [11].

4. Modifications in the microstructure of alloy by thermo-mechanical treatment

Thermo-mechanical treatment, is a metallurgical process, which involves in combining both the mechanical or plastic deformation process (compression or forging, rolling etc.,) and the thermal processes (heat-treatment, water quenching, heating and cooling at various rates) into a single process.

4.1 Thermal processing during the manufacturing of alloy

The mechanical behaviour of NiTi alloy is determined by the relative proportions and characteristics of the microstructural phases. Heat-treatment (thermal processing) is a fundamental approach towards regulating the transition temperatures of NiTi alloys and affecting the fatigue resistance of NiTi endodontic files. More the

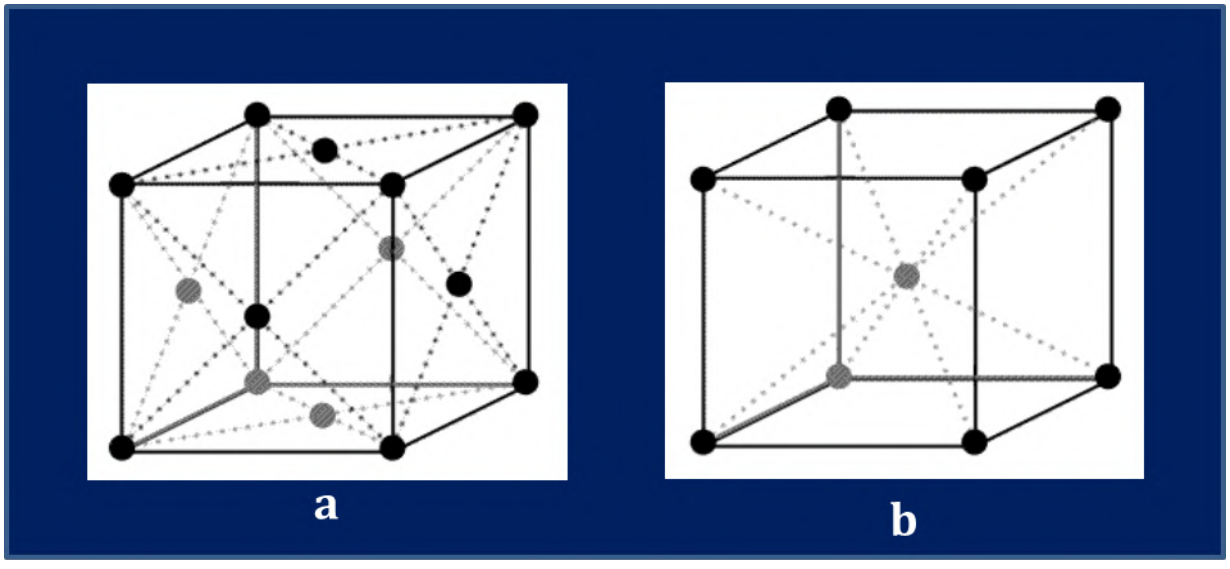

Figure 2. Crystalline structure changes from the cryogenic treatment. Where; a. Before treatment: Face-centered cubic austenitic structure, and b. After treatment - Cell-centered structure

martensitic NiTi alloy more is the flexibility and fatigue resistance of an instrument as it produces a better arrangement of the crystal structure and changes in the relative percentage of phases present in the alloy as found by De Vasconcelos [12]. Development of the next-generation endodontic instruments is made on the enhancements in these areas of material management.

4.2 M-Wire

M-Wire was developed to produce superelastic NiTi wire blanks that contain substantial stable martensite under clinical conditions M-Wire was developed. Martensite, stress-induced martensite (SE), and austenite are three different forms of NiTi. Softness, ductility and easy deformation are observed when the material is in its martensite form. The austenitic NiTi is strong and hard, whereas SE NiTi is highly elastic. The martensitic phase transformation has excellent fatigue resistance because of the energy absorption characteristics of its twinned phase structure. In 2007, M-wire (Dentsply Tulsa- Dental Specialties, Tulsa, OK, USA) introduced and it contains portions that are in both the deformed and micro twinned martensitic, pre-martensitic R-phase, and austenite while maintaining a pseudoelastic state [13]. The austenite-finish temperature (A_f) of M-Wire is in the range of 45^0C–50^0C [14]. This temperature range indicates that the instruments manufactured from M-Wire would be necessary for the martensitic phase at room temperature. Various M-Wire instruments include Dentsply's ProFile GT Series X, ProFile Vortex, and ProTaper Next files, Path Files, WaveOne and Reciproc (VDW, Munich, Germany). Gao *et al.* [15] studied the effect of cyclic fatigue loading on M-wire and a regular SE wire at two different rational speeds. They found multiple crack-initiation sites with more than 50% of broken files, which are made from SE wire and a single crack initiation on files made of M -Wire.

4.3 R-Phase

R phase is formed during the forward transformation of martensite to austenite on heating and reverse transformation from austenite to martensite on cooling. Upon heating, martensite will start transforming to R-phase at R_s temperature, and this transformation will be finished at the R_f temperature. On further heating, R-phase starts transforming to austenite at the A_s temperature, and transformation is finished at A_f temperature. If heated above A_f temperature, it will be converted entirely to austenite. Then, upon cooling to a sufficiently lower temperature, the alloy starts transformation from austenite to R-phase at the R_s temperature and this transformation will be finished at the R_f temperature. By further cooling, the R-phase starts transforming to martensite at Ms temperature and finished at M_f (Figure 3) [16]. The alloy obtains greater strength and a lower modulus of elasticity on comparison with stainless steel. Therefore, instruments made with R-phase wire are more flexible than stainless steel [17]. Twisting of the wire can be performed once the R-phase is identified as it optimises the grain structure in the metal. The grinding process weakens the metal's structure at the molecular level that results in creating microfractures on its surface, leading to fracture of files [18]. In 2008, SybronEndo (Orange, CA, USA) developed Twisted Files (TF) and K3XF files by twisting the intermediate alloy followed by a heat-treatment process. R-phase exhibits lower shear modulus compared to martensite and austenite phases. Further, the transformation strain is also less than one-tenth to that of martensitic transformation. Instruments made with R phase are fully austenitic at ambient and body temperatures [19] and also imparts greater flexibility and increased resistance to flexural fatigue as stated by recent reports [20]. On the contrary, Park *et al.* [21] observed that this manufacturing method fails in providing any beneficial effect with regard to torsional fracture.

4.4 Controlled memory NiTi alloys (CM Wire)

CM Wire (DS Dental, Johnson City, TN) was introduced in 2010. It is a novel NiTi alloy with flexible properties. These CM wires are manufactured by a proprietary thermo-mechanical process, which allows the instruments to be pre-curved before they are placed into the root canals. In addition, this process also increases the flexibility, the transformation temperatures (A_f to about 50°C), reduces the shape memory, and also helps in obtaining stable martensite at the body temperature. However, they revert to their original shape on sterilization. Various CM NiTi file systems include Hyflex CM (ColteneWhaledent, USA), Typhoon CM (Clinician's Choice Dental Products, USA) and ProFile Vortex Blue (Dentsply).

During clinical use, the conventional NiTi files are in the austenite phase as their A_f temperature is at or below room temperature. However, the A_f of CM files is certainly above the body temperature, which results in the formation of both martensite and R-Phases in addition to the austenite phase. The Hyflex CM and Typhoon CM instruments contain a combination of martensitic R-phase

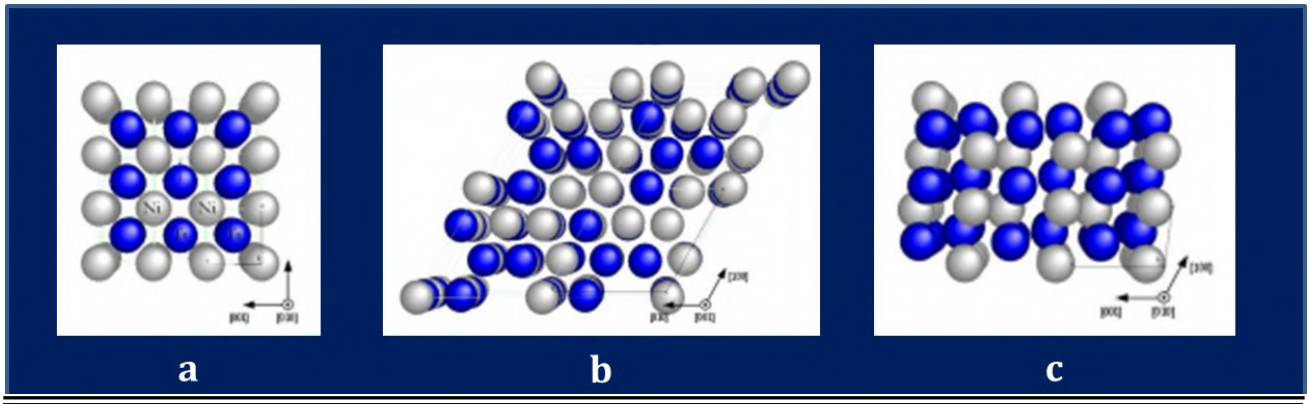

Figure 3. Experimental structures of NiTi alloys. a. B2 austenite PM3-M, b. R-phase P3, and c. B19 martensite p21/m (cubic coordination); Where grey and blue coloured spheres are Ni and Ti atoms respectively.

and austenitic phase at body temperature as their A_f temperatures are slightly above the body temperature [22]. Shen *et al.* [23] reported that instruments made from CM wires exhibited around 300% to 800% more resistance to fatigue failure compared to instruments made from conventional NiTi wires. Longer fatigue life observed with the square configuration of NiTi instruments made from CM Wire than the triangular configuration.

4.5 Thermal processing after machining of files / Post-machining heat-treatment

Thermal processing used to overcome the defects occur during the machining process and also to modify the crystalline structure of alloys. Thermocycling of NiTi alloys causes the martensitic transformation to occur in two stages instead of in a single stage. The stage-1 transformation (A-M) takes place in Ni-rich NiTi alloys. Stage-2 transformation (A-R-M) takes place after the additional heat-treatment, which precipitates finely dispersed Ti_3Ni_4 particles in the austenitic matrix. Accordingly, the R-phase is formed instead of martensite due to the presence of fine dispersed Ti3Ni4 particles. Therefore, it necessitates additional cooling of alloy to form martensite and hence, martensitic transformation occurs in 2 steps (A-R-M) [24].

4.6 Vortex blue

Vortex blue is a newly developed NiTi rotary instrument with improved fatigue resistance, cutting efficiency, flexibility, and canal centering capability, and that is made from M-Wire [25]. Its A_f temperature is around 38°C. Vortex blue has a 2-stage transformation, as observed in studies. Unique "blue colour" is seen by Vortex Blue instruments on comparison with traditional SE NiTi instruments. Proprietary manufacturing process leads to the "blue-colour" oxide surface layer of Vortex Blue files. Compensation for the loss of hardness along with improvement in the cutting efficiency and wear resistance is achieved by the relatively hard titanium oxide surface layer on the Vortex Blue instrument.

4.7 ProTaper Gold (PTG)

The geometries of both ProTaper Gold (PTG) ProTaper Universal (PTU) are the same with a convex triangular cross-section and progressive taper. The files are heat-treated after their manufacturing at around 370-510°C for a variable period. Similar to CM wire, these files exhibit two stage specific transformation behaviour and high A_f temperature about 50°C [26] that offers increased resistance to cyclic fatigue loads for PTG instruments compared to instruments made with PTU. Less shape memory is observed with PTG than NiTi so unopened package of files exhibiting a slight degree of curvature is not a surprising feature. It is an advantage rather than a defect as supposed by the manufacturer. The file follows the root canal anatomy being shaped upon removal from a curved canal.

4.8 WaveOne Gold

Unique heat-treatment before and after file manufacturing led to the development of WaveOne Gold. The SE NiTi alloy is subjected to a unique heat treatment process in a temperature range of about 410° to 440°C, under constant strain (3-15 kg). The finished instrument is subjected to a second heat treatment process in a range of 120°C to 260°C, after machining the working portion of the file. The A_f temperature of WaveOne Gold is in the range of 40°C-60°C.

Numerous manufacturers suggested that this technology improves the flexibility and strength of the instrument. Torsional resistance could be enhanced due to off-centred parallelogram-shaped cross-section design.

4.9 Hyflex EDM

Coltene Whaledent manufactured Hyflex ED based on the EDM method. EDM stands for electrical discharge machining (EDM), which is a noncontact thermal erosion process, that is used to machine electrically conductive materials with the help of controlled electrical discharges. This process generates the electrical sparks, which results in a local melting and partial evaporation of small portions of material resulting in the formation of a typical crater-like surface finish. Then, the instrument is cleaned through ultrasonic in an acid bath, followed by a heat treatment process at a temperature ranging between 300-600°C for 10 min to 5 hours before or after the cleaning process. A_f temperatures over 52°C are observed with EDM files [27]. Early material failure is avoided by producing a non-directional surface finish by EDM process.

On comparison with other rotary NiTi instruments, HyFlex may be up to 300% more fatigue- resistant as reported by manufacturer. The instrument regains its shape by sterilization. Peters *et al.* [28] reported that more than half of the plastically deformed instruments recovered to their original shape during sterilization. However, sterilization had shown no effect on the small instruments in retaining their shape. Therefore, care should be taken regarding reuse of small HyFlex rotary instruments. Less apical pressure against canal walls is required to be applied than with conventional SE NiTi files of the same size and taper.

4.10 K3 XF

In 2011, SybronEndo developed K3XF by taking advantage of R-phase technology. Fabrication takes place by a grinding process rather than a twisting process. A Special heat treatment process is performed on K3XF files after the grinding process to enhance flexibility and strength. This heat-treatment also modifies the crystalline structure of the alloy to accommodate some of the internal stress caused by the grinding process [29]. K3XF instruments undergoing post-machining heat treatment differs them from K3, but both are identical in shape. Since K3XF instruments have an A_f temperature below 37°C therefore, it has an austenite structure at body temperature and exhibits superelastic property during clinical application. The heat treatment processing used for K3XF modifies the transformation temperature by releasing crystal lattice defects and diminishing internal strain energy. K3XF instruments have numerous micropores with various diameters on the surface of the instrument flute. These small pores serve as a local stress/strain discontinuity from which the crack nucleates and does not contribute to the failure [30].

4.11 XP Endo Shaper

FKG Dentaire, La-Chaux-de-Fonds, Switzerland introduced XP endo shaper in 2016 with 0.30 diameter and 0.1 taper that could expand to 0.4 taper. At room temperature, these instruments are relatively straight in their M-phase (martensitic state) and change to a curved shape on exposed to intracanal temperature due to a phase transformation to A-phase (austenitic state). The martensite to the austenite phase occurs naturally in the body temperature (32°C and 37°C) with Af temperature at 35°C. In a dynamic state, the instrument has a twisted shape, with several twists twisted along its length. Shape memory effect is exhibited when these instruments are inserted into the root canal (M-phase to A-phase) and possess superelasticity during preparation. The curved shape enables the preparation of complex root canal morphologies with the potential to adapt to canal irregularities.

5. Conclusion

Thermomechanical treatment of NiTi alloy allows a transition in the phase composition leading to the appearance of martensite or R-phase under clinical conditions. While M-Wire and R-phase instruments maintain an austenitic state, CM Wire, as well as the Gold and Blue heat-treated instruments are composed of substantial amounts of martensite. High torque values at fracture are revealed by austenitic instruments possessing superelastic properties. Thus, to shape straight or slightly curved root canals, these files are appropriate. Use of austenitic alloy in pathfinding instruments compensates for the decreased torque resistance caused by the smaller diameter of these files. Martensitic instruments are more flexible with enhanced resistance to cyclic fatigue, and they also reveal a greater angle of rotation but lower torque at fracture due to an increased amount of the martensite phase. In complex curvatures and root canal anatomies, cyclic fatigue is known to occur more likely. Thus, in cases of severely curved root canals or those with double curvature martensitic instruments should be preferred. Moreover, when trying to bypass ledges, Martensitic instruments are useful.

Conflicts of interest: Authors declared no conflicts of interest.

Financial support: None

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Review Article International Journal of Dental Materials 2020; 2(2) ISSN: 2582-2209

Self-sealing resin fixators in dentistry

Rama Krishna Alla[1,*], Vineeth Guduri[2], Savitha P Rao[3], Suresh Sajjan MC[4], Ramaraju AV[4]

[1]Assistant Professor, Department of Dental Materials, Vishnu Dental College, Bhimavaram, West Godavari, Andhra Pradesh, India—534202.

[2]Reader, [4]Professor, Department of Prosthodontics and Implantology, Vishnu Dental College, Bhimavaram, West Godavari, Andhra Pradesh, India—534202.

3Assistant Professor, Department of Dental Materials, VS Dental College and Hospital, Bengaluru, Karnataka, India.

INFORMATION

Article History

Received *11th May 2020*
Accepted *17th May 2020*
Available online
31st May 2020

KEYWORDS

Luting agents
Resin-based cements
Self-adhesive resin cements
Dual-cure
Micromechanical adhesion
Bis-GMA
UDMA
TEGDMA
Self-etch
Conditioning of dentin

ABSTRACT

Fixed indirect restorations bond to the prepared tooth surfaces with the use of a variety of luting agents depending upon the purpose of that rehabilitation. Success and failures of these restorations have been attributed to the quality of their bond with the tooth substrate. However, the advent of resin-based and self-adhesive resin luting agents have greatly changed this equation by altering the conventional bonding mechanisms and the durability of bond. The limited literature details of these self-adhesive resin luting agents require further exploration for the benefit of dental professionals. This review provides an overview of the composition, chemical interactions, favourable and unfavourable properties to be known for improving the scope of their utilization in dentistry.

1. Introduction

Rehabilitative appliances fabricated to restore the missing tooth/teeth include removable prostheses and fixed partial dentures (FPD). FPDs mandate preparation of teeth surfaces to a thickness of 1.5 to 2mm involving both enamel and dentin to accommodate the bulk of prosthetic materials and a thin bonding space. This space and the substances filling it determine the quality of bond with natural tooth/ teeth and thereby govern the success and durability of FPDs. Dental cements, commonly referred to as 'luting agents', are widely used adhesive agents between fixed partial dentures and the tooth structure. These luting agents not only provides bonding between the FPD and the tooth structure but also prevent the formation of secondary caries and penetration of oral fluids into the prepared surface and insulate the thermal conduction by filling the gap between the tooth surface and the restoration. Further, dental cements are used to bond orthodontic appliances to the teeth and cementing pins and posts to retain dental restorations. Fixed orthodontic appliances are attached to the natural tooth surface without any reduction [1, 2].

Various luting cements used for luting of indirect restorations and orthodontic appliances are zinc phosphate, zinc polycarboxylate, glass ionomer, hybrid ionomer, resin-modified glass ionomer and polyacid modified resin cements [1, 3-6, 11-13]. A group of resin-based cements have been developed with enhanced bonding mechanisms by using the acid-etch technique for adhering to enamel and potential bonding molecules for attaching to conditioned dentin with an organic or inorganic acid. These resin-based cements are used for luting orthodontic

Correspondence: *Corresponding author Email Address: ramakrishna.a@vdc.edu.in

How to cite this article: Alla RK, Guduri V, Savitha P Rao, Suresh Sajjan MC, Ramaraju AV. Self-sealing resin fixators in dentistry. Int J Dent Mater 2020;2(2): 60-68.
DOI: http://dx.doi.org/10.37983/IJDM.2020.2205

brackets or resin-bonded bridges [4]. This article reviews the composition, chemistry, properties, advantages, and disadvantages of resin-based luting cements with more emphasis on self-adhesive resin cements.

2. Resin-based luting agents

Resin cements were introduced in the mid-1980s. Biomer was the first resin cement marketed by Dentsply/ Caulk, in 1987. Resin cements contain resins or polymers as the primary reactive ingredients and to which fillers have been added to modify the coefficient of thermal expansion (CoTE) and water sorption thereby increasing the strength and hardness of polymers [1,4]. These resin-based cements are also possessing anti-cariogenic property as they contain fluoride agents [14]. These resin-based cements are widely used for luting of non-metallic restorations, resin-bonded FPDs, porcelain crowns and veneers, ceramic and resin composite inlays and onlays [1,4]. However, these early resin-based cements do not chemically adhere to enamel and dentin, leading to microleakage and also possess high film thickness. Besides, they cause pulpal irritation due to leaching out of residual monomer [15], and also undergo discolouration due to high residual amine levels after polymerization [1,4,16]. It was reported that the resin -based cements with a dentin bonding agent exhibited superior retention of crowns on teeth compared to using zinc phosphate cement [1,4].

Later, aromatic dimethacrylate-based resin cements, bis-GMA based, have been developed [1,7]. Bis-GMA resin is a multi-functional methacrylate resin developed by Dr Bowen. The bis-GMA (2,2-bis[4-(2 hydroxy-methacryloxypropoxy) phenyl] propane) resin can be described as an aromatic ester of dimethacrylate, synthesized from an epoxy resin and methyl methacrylate [1,4,17]. Bis-GMA is extremely viscous at room temperature; hence, a diluent resin, such as triethylene glycol dimethacrylate (TEGDMA) is blended with it to reduce the viscosity. Resin cements are available as powder/liquid, encapsulated, or paste/paste systems and are classified into three types based on the method of polymerization as chemical-cured, light-cured and dual-cured [1,4,17].

3. Self-adhesive resin cements

Self-adhesive resin cements were introduced in 2002 to overcome some of the disadvantages of both conventional (zinc phosphate, polycarboxylate, and glass-ionomer cements) and resin cements [18].

Self-adhesive resin cements have a wide range of clinical applications as they have favourable characteristics of conventional luting and resin cements [18,19]. They possess good esthetics, best mechanical properties, good dimensional stability, and micromechanical adhesion as shown wit resin cements. Unlike conventional resin cements, no pre-treatment of the tooth surface is required prior to luting with self-adhesive resin cements. The application procedure of self-adhesive resin cements is simple and is accomplished in a single clinical step, similar to the application procedures used with zinc-phosphate and polycarboxylate cements. Furthermore, patients do not experience any postoperative sensitivity as the smear layer is not removed. They are also moisture tolerant compared to the earlier luting agents. These self-adhesive resin cements also exhibit anti-cariogenic properties as they release fluoride ions in a manner comparable to glass ionomer cements [18-20].

3.1 Composition of Self-adhesive resin cements

Self-adhesive resin cements are usually dispensed in individual syringes. The most popular dispensing system is a two-paste system with dual-barrel syringe dispensers. One paste contains the predominant functional acidic monomers, conventional di-methacrylate monomers (e.g., bis-GMA, UDMA, and TEGDMA), and initiator systems for both light and self-cured reaction. The other paste contains fillers like fluoro-alumino-silicate, silanated barium glasses, or both, and silanated silica particles. Further, this paste also contains activator-initiator systems, and methacrylate monomers [4,20, 21]. The composition of various contemporary materials was described in table 1.

3.2 Setting reaction of self-adhesive resin cements

The self-adhesive resin cements undergo a free-radical addition polymerization, which is either self-activated or dual-cured. The initial pH is low and it is necessary for the adhesion mechanism. The acidity is neutralized by the reaction between phosphoric acid groups and the alkaline glass as the polymerization reaction proceeds [20,21]. Currently available self-adhesive resin cements are dual-cure resin materials, which depends on both light and self-curing mechanisms [16]. Literature reported that the overall degree of conversion of dual-cure resin cements might be compromised as the self-curing mechanism is relatively slow, and it can be interrupted by the formation of a first polymer network, which is triggered by light activation [22-24]. The set material is mainly a cross-linked polymer, which is covalently bonded with silane coupling agents.

Table 1. Self-adhesive resin cements and their characteristics

Product	Delivery system	Working and setting time	Shades	Composition
BisCem® (Bisco, Schaumburg, IL, USA)	Paste/paste dual syringe; direct dispensing through a mixing tip.	1min/6min at 22ºC	Translu-cent Opaque	Bis (hydroxyethyl metha-crylate) phos-phate (base), tetraethylene glycol di-methacrylate, dental glass.
BeautiCem SA, Shofu Inc, Japan	Paste/paste dual Auto-mixing sy-ringe; direct dispensingthrough a mix-ing tip and also available for manual mixing material			
Bifix SE, Voco, Ja-pan	Dual curing system	20 sec – light curing	3 shades	Glycerine dimethacrylate-based resin
Breeze™ (Pentron Clinical Technolo-gies, Wallingford, CT, USA)	Paste/paste dual syringe; direct dispensing through a mixing tip	1min/4min at 22ºC	A2 Translu-cent Opaceous White	Mixture of Bis-GMA, UDMA, TEGDMA, HEMA, and 4-MET resins, silane-treated barium borosilicate glasses, silica with initiators, stabilizers and UV absorber, organic and/or inorganic pigments, opac-ifiers
Calibra Universal, Dentsply, Milford	2-paste system	10 sec/45 sec		
Clearfil SA (Kurar-ay, Tokyo, Japan)	Dual-barrel syringe	1min/5min	A2 White	Bis-GMA, TEGDMA, MDP, barium glass, silica, sodium fluoride
Embrace WetBond resin cement (Pulp-dent; MA, USA)	Automix or standard syringe packaging	Completely autocures in 7min	One shade	Di-, tri-, and multi-functional acrylate monomers into a hydrophilic, resin acid-integrating network (RAIN).
G-Cem™ (GC; Tokyo, Japan)	Capsules	2min/4min	A2, AO3, Translu-cent, BO1	Powder: fluoroaluminosilicate glass, initiator, pigment. Liquid: 4-Met, phosphoric acid ester monomer, water, UDMA, dimethacrylate, silica powder, initiator, stabilizer
G-Cem LinkAce, GC America, USA	dual-cure self-adhesive resin deliv-ered in double barrel automix syringe		A2, Trans-lucent, Opaque (AO3) & (BO1)	
iCEM® (Heraeus Kulzer)	Double syringe			
Maxcem Elite ™ (Kerr; Orange, CA, USA)	Paste/paste dual syringe; direct dispensing through a mixing tip	2min/3min	Clear White White opaque Yellow Brown	GPDM (glycerol dimethacrylate dihydro-gen phosphate), comonomers (mono,di, and tri-functional methacrylate mono-mers), proprietary self-curing redox activator, photo-initiator (camphor-quinone), stabilizer, barium glass fillers, fluoroaluminosilicate glass filler, fumed silica (filler load 67%wt, particle size 3.6µm
Multilink Sprint Ivoclar Vivadent, Schaan, Lichten-stein	Paste/paste dual syringe; direct dispensing through a mixing tip	Working time : 130±30s, Setting time: 270±30 s (based on oral temperature)	Transpar-ent Yellow Opaque	Dimethacrylates and acidic monomers. The inorganic fillers are barium glass, ytterbium trifluoride and silicon dioxide. The mean particle size is 5 µm. The total volume of inorganic fillers is pprox.. 48 %

Contd…,

Table 1. Self-adhesive resin cements and their characteristics

Product	Delivery system	Working and setting time	Shades	Composition
Monocem™ (Shofu Dental; San Marcos, CA, USA) Panavia SA, Kurar-ay Noritake Dental	Paste/paste dual syringe; direct dispensing through a mixing tip	Unlimited working time (7min in anaerobic conditions)	Translucent Bleach white	
RelyX™ Unicem (3M ESPE; St Paul, MN, USA) RelyX™ Unicem 2 (3M ESPE; St Paul, MN, USA)	Capsules (Aplicap: 0.001ml; Maxicap: 0.36ml)	2min/5min at 22ºC	A1, A2, Universal Translucent, White opaque A3 Opaque	Powder: glass fillers, silica, calcium hydroxide, self-curing initiators, pigments, ligth-curing initiators. Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiatirs, ligth-curing initiators
SeT (SDI, Australia; SE)	Capsules	5min	Translucent, A1, A2, AO3 White opaque	UDMA, phosphate, fluoroaluminosilicate glass, silica.
SmartCem® (Dentsply-Caulk- Germany)	Dual-barreled syringe	2min/6min	Translucent Light Medium Dark Opaque	Urethane dimethacrylate; di- and trimethacrylate resins; phosphoric acid modified acrylate resin; barium boron fluoroaluminosilicate glass; organic peroxide initiator; camphorquinone photoinitiator; phosphene oxide photoinitiator; accelerators; butylated hydroxy toluene; UV stabilizer; titanium dioxide; iron oxide; hydrophobic amorphous silicon dioxide.
SpeedCEM™ (Ivoclar, Vivadent)	Double syringe	Working time: Self-cure: 100 – 140 Seconds, Dual-cure: 100 – 140 seconds S etting time: (37 °C) Self-cure: 150 – 220 seconds, Dual-cure: 150 – 220 seconds	Transparent Opaque Yellow	Dimethacrylates, ytterbium trifluoride, co-polymer, glass filler, silicon dioxide, adhesive monomer initiators, stabilizers and pigments

Formation of ionic bridges between carboxylic groups and ions released by the glass may also be observed [20].

3.3 Properties

Self-adhesive resin cements are not biocompatible due to their higher cytotoxicity compared to resin and acid -base cements. However, dual-cure self-adhesive resin cements possess reduced cytotoxicity [20]. Also, the degree of polymerization of dual-cure adhesive resin cements is less. de Souza Costa CA *et al.* (2006) [25] demonstrated no pulpal response with RelyX Unicem cements even after 60 days of their placement. It can be attributed to its chemical adhesion to tooth structure, low solubility, and a self-neutralizing mechanism during the polymerization reaction. On the other hand, Variolink II with the bonding agent, Excite demonstrated severe effects on the pulp-dentin complex.

These cements contain acidic functional monomers, which can neutralise or interfere with the free radicals and retard the polymerization reaction. This delayed polymerization can last from 24 hours to 7 days [26]. The rate of polymerization merely depends on the ratio of self-curing to light-curing components, light exposure time, intensity of the light source, the type and the thickness of the restorations [21]. Also, neutralization of the acidic monomers can significantly affect the rate of polymerization. The presence of residual acidity of these monomers may reduce the curing rate and prolong the final set of the cement. These harmful effects could result in cement with increased water sorption. Storage temperature is another factor that influences the polymerization of self-adhesive resin cements. If the cement is stored at temperatures higher than the room temperature would have a deleterious effect on the curing rate. It is well known that the rate addition polymerization is directly proportional to the temperature. The higher the temperatures, the faster will be the rate of polymerization reactions. However, it may also be influenced by the individual components of the cement and their response to the temperature [27]. The ideal temperature to store self-adhesive resin cements is in the range of 4^0C to 18^0C, and it is necessary to bring them to room temperature before using [21].

Polymer-based cements tend to absorb water, resulting in swelling of the cement. This increase in size may be considered as an advantage as it compensates the polymerization shrinkage and improves the marginal seal. However, excessive swelling may not be desirable as it creates more stress at the interface of the cement and the restoration. Generally, the solubility of the polymer-based cements in water is very less. The rate of sorption and solubility depends on the type of the resin matrix that is present in the cement [28-30], the amount of residual hydrophilic components in the set matrix [30], cross-linking density and porosity [31], and amount of residual acidic monomers and type of polar functional groups [31–33].

These cements are initially hydrophilic in nature. The low pH, along with high hydrophilicity, helps in proper wetting and providing bonding to the tooth substrate. The acidic functional monomers are slowly neutralised as they start their chemical reaction. Then during demineralization, the pH of the functional acidic monomers is slowly neutralized with the hydroxyapatite and filler particles. As the pH increases, the material becomes more hydrophobic and becomes less susceptible to hydrolysis [34,35]. The film thickness of these cements is between 15 and 20 μm.

The mechanical properties of self-adhesive resin cements are superior compared to the conventional luting agents and less than the resin cements. However, they vary among commercial materials. It was reported that the light-activated adhesive resin cements exhibit better mechanical properties than the self-cure cements. Kumbuloglu *et al.* (2004) [36] reported more compressive strength and hardness with the RelyX Unicem light-curing cements compared to RelyX ARC, Panavia F, and Variolink cements. On the contrary, Piwowarczyk A *et al.* (2003) [37] reported more compressive and flexural strengths with the three cements than the RelyX Unicem. No significant differences were observed in the fatigue strength and resistance to the fracture among the various commercial materials irrespective of their curing mechanisms [38 - 41].

3.4 Bonding mechanisms

Self-adhesive resin cements do not require the application of a separate adhesive before cementation. However, the performance of various self-adhesive resin types of cement can be improved by additional surface treatments before cementation [42–44].

3.4.1 Bonding with enamel and dentin

Self-adhesive resin cements adhere to the tooth structures via micromechanical interlocking and chemical interaction between the acidic groups and the hydroxyapatite groups in the teeth [20]. On cementation, self-adhesive resin cements first demineralize the tooth substrate and then infiltrate enamel and dentin. However, they interact only superficially with dental hard

tissues [18, 45,46]. The bond strength of these cements with the natural tooth substrates are more compared to the glass ionomer cements and comparable to that of the self-etching adhesives. Therefore, self-adhesive resin cements may be considered as an alternative material to glass-ionomer cement for cementation of metal-based and high-strength ceramic restorations [18].

The acidic monomers of self-adhesive resin cements provide lower interprismatic hybridization as they are weaker compared to the traditional phosphoric acid etchants. Therefore, enamel may not be effectively demineralized that resulted in weak bond strengths with enamel compared with conventional hybridization techniques that are usually seen with the separate etching and bonding approach [47].

Pre-etching the dentin with phosphoric acid may not provide adequate bonding with self-adhesive resin cements as it results in inadequate resin infiltration into the exposed collagen fibril network [47, 48]. Numerous studies have reported that the use of polyacrylic acid instead of phosphoric acid gives better results, especially with the concentration of 10-25% and at low pH [49,50]. Thermocycling or aging of the restorations in different conditions may also reduce the bond strengths with enamel and dentin [51].

3.4.2 Bonding with the restorative materials

Self-adhesive resin cements not only adheres with the natural tooth but also with ceramics and some metals and alloys.

3.4.2.1 Ceramic restorations

Ceramics are the group of widely used esthetic indirect restorative materials [52,53]. A suitable luting agent such as glass ionomer cements and resin cements may be used to seat the ceramic restoration firmly on the prepared tooth [1]. The durability of ceramic restoration depends on the quality of bonding by the luting agent [54]. In addition, the aesthetics of ceramic restorations also depend on the type of luting agent is used.

In general, the internal surface of the ceramic restorations is treated to enhance the bond strength with the luting agent. These treatments include sandblasting or etching the internal surface before the cementation. Silicate-based ceramics achieve good bonding with the self-adhesive resin cements by two simultaneous mechanisms. They include micromechanical retention, which is provided by acid-etching of the ceramic surface, and followed by the chemical coupling with the help of a silane coupling agent [55-60].

Usually, hydrofluoric acid gels are used to etch the surface of the ceramics, followed by silanization [61,62]. The hydrofluoric (HF) acid reacts with the silica that is present in the glassy matrix results in dissolving the surface to the depth of a few micrometres [56] and exposes the crystalline structure [57]. Then, a bifunctional silane coupling agent is applied that promotes a chemical interaction between the silica in the glass phase of ceramics and the methacrylate groups of the resin cement through siloxane bonds [56, 63-65]. Silanization reduces the contact angle and increases the wettability of the ceramic surface [66], making it a suitable substrate for bonding with resin cements. It has been shown that the light-cured cements give better results on cementing the ceramic veneers compared to self-cured resins.

The colour stability of self-cure resins cements is poor as they contain amines activators, which tends to discolour the ceramic veneers.

Shear bond strength has been improved when self-adhesive resin cements are used in conjunction with sandblasted (aluminum oxide) zirconia ceramic restorations [67]. It was also reported that the performance of the zirconia crown cemented with self-adhesive resin was improved when the internal surface of the restoration is pre-treated with light-pressure sandblasting followed by the application of MDP-containing primers [68-71].

Various systematic reviews suggested that physicochemically conditioned zirconia crowns combined with MDP-based self-adhesive resin cements exhibit favourable results on adhesion with each other [69,70]. Numerous studies reported that the thermocycling improved the shear bond strength with self-adhesive resins cements [72].

3.4.2.2 Bonding to endodontic posts

Self-adhesive resin cements show significantly higher push-out strength to fibre posts compared to zirconia posts [73]. However, limited research was done in this area.

3.4.2.3 Bonding with Titanium abutments

Self-adhesive resin cements exhibited significantly higher with titanium abutments compared with zinc phosphate and glass ionomer cements. However, the bond strength values achieved with the self-adhesive resin cements were incomparable to retention achieved using polycarboxylate cement [74].

4. Conclusion

Self-adhesive cements are promising luting agents and a viable clinical alternative material in indirect restorative procedures due to their simplified technique, reduce the occurrence of postoperative sensitivity and are suitable for a wide range of applications. They can be extensively used for cementation of fibre posts, monolithic zirconia crowns, and PFM crowns when moisture control is challenging for adhesive application. Based on the literature available, RelyX™ Unicem was the most investigated self-adhesive cement and proved to be satisfactory and comparable to other multistep resin cements. However, long-term studies are necessary to evaluate the clinical performance of self-adhesive resin cements prior to making any general recommendation regarding their use.

Conflicts of interest: Authors declared no conflicts of interest.

Financial support: None

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International Journal of Dental Materials

Volume 2 Number 2 April-May 2020

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