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International Journal of Dental Materials (e-ISSN: 2582-2209) welcomes editorial queries, original studies, evidence based research works and practical innovations, reviews, case reports and concise communications. This journal intends knowledge transfer and spread of verified information from valuable researchers to all fellow dental fraternity. Manuscripts showcasing studies on dental biomaterial properties, performance, induced host response, immunology and toxicology will attain the highest priority for publication. Documentation emphasising advancing dental technology, innovations in dental materials design and their clinical viability succeed the hierarchy of publishing preference.

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Comparative evaluation of sealer penetration depth into radicular dentinal tubules using confocal scanning microscope: an *in vitro* study

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ABSTRACT

Background: Endodontic treatment involves the removal of the vital and necrotic contents of the root canal through chemo-mechanical means followed by obturation of the prepared root canal to prevent the ingress of fluids and avoid bacterial infection or regrowth. Root canal sealers and core filling materials are used together to fill the irregularities in the root. Penetration into the dentinal tubules also results in the inhibition of bacterial regrowth and increases the success of root canal therapy.

Aim: This study aimed to evaluate the penetration depth of various sealers into the dentinal tubules using a confocal microscope.

Materials and methods: A total of 65 specimens were decoronated to standardize the root length of 13mm. Working length was determined, and Biomechanical preparation for all the samples was done with a rotary ProTaper file till F4. Samples were randomly divided into five groups containing 13 teeth in each group based on the sealer used, namely Group 1: Endomethasone (n=13), Group 2: AH-Plus (n=13), Group 3: Roekoseal (n=13), Group 4: MTA Fillapex (n=13), Group 5: Endosequence BC (n=13). All the sealers were labelled with Rhodamine -B dye, and samples were obturated using cold lateral compaction technique. The specimens were sectioned orthogonally at coronal, middle, and apical thirds. All the samples were examined with a Zeiss Pascal Laser Scanning Microscope to examine the sealer penetration depth into the dentinal tubules. The data were subjected to statistical analysis using one- way Analysis of Variance (ANOVA) and Tukey's Honest Significant Difference (HSD) tests.

Results: Endosequence BC showed the highest penetration into dentinal tubules, followed by MTA Fillapex and Roekoseal, AH-Plus, and Endomethasone exhibited the least penetration.

Conclusion: Endosequence BC sealer exhibited maximum penetration. All the groups showed maximum penetration at coronal third, followed by the middle and apical third.

1. Introduction

Pulpal and periapical diseases are primarily related to microorganisms and their by-products in the root canal system, which occurs due to the invasion of bacteria through caries or fracture. The main objective of endodontic treatment is to

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eliminate microorganisms from the root canal space and also prevent it from reinfection [1]. Chemo-mechanical preparation is considered the most effective step in the management of the infected root canal space, followed by 3-dimensional obturation with a biocompatible material [2]. Hence, there is a need to obturate the root canal space thoroughly to prevent leakage and to entomb residual debris and recalcitrant bacteria. Obturation eliminates all avenues of leakage from the oral cavity and the periradicular tissues into the root canal system by creating a fluid-tight seal. Root canal sealers are used along with a core-filling material to attain an impervious seal between the core material and root canal wall [2,3]. Commercially there are many sealers available in clinical practice. Variations in the mechanical and chemical properties of sealer cement also influence the depth of penetration [4]. Therefore, it is essential to compare the penetrability of various sealers that are used in routine clinical practice. Endomethasone N is a zinc oxide eugenol sealer with anti-inflammatory activity due to the presence of hydrocortisone acetate. AH Plus, which is resin-based cement has excellent mechanical properties and low polymerization shrinkage. RoekoSeal is a silicon-based sealer with no shrinkage and excellent sealing property. MTA Fillapex is MTA based sealer with tissue recovery property and a lack of inflammatory response. Endoseqence BC sealer which is a bioceramic sealer which sets in the presence of moist dentine. Hence this in vitro study aimed to evaluate the penetration efficiency of five different sealers into dentinal tubule using Rhodamine B dye under confocal laser scanning microscopy.

2. Materials and methods

A total of 65 extracted human mandibular premolars with a single root and single canal were included in the study. For standardization, all the samples were decoronated to a length of 13mm by using a double-faced diamond disc (KG Sorensen, Barueri, SP, Brazil). Pulpal tissue extirpation and working length were determined. Biomechanical preparation for all the samples was done in crown-down motion using ProTaper rotary nickel-titanium files (Dentsply Maillefer). Canals were irrigated between the use of files with 5ml of 3% Sodium hypochlorite (Prime dental PVT LTD., India). To remove the smear layer, all canals were irrigated with 3mL of 17% ethylenediamine tetraacetic acid (DESmear, Anabond Stedman Pharma research, India). Final rinse performed by using 5 mL of distilled water to remove any remaining irrigating solution. All the irrigation procedure was followed using a side vented needle placed 1mm short of the apical foramen. The canals were dried with sterile absorbent paper points (Prime dental PVT LTD., India.) after irrigation. All intracanal procedures were done by a single operator to eliminate inter-operator variability. Teeth were then randomly divided into five experimental groups using computer-generated sequence allocation, consisting of 13 teeth in each (n=13) sealer group. Sealers used in this study were Endosequence (Brasseler USA, Savannah, GA), MTA-Fillapex (Angelus, Londrina, Brazil), Roekoseal (Coltene/Whaledent, Langenau, Germany), AH-Plus (Dentsply-Maillefer, Tulsa, OK, USA), Endomethasone (Septodont, Saint-Maur, France). Rhodamine B dye was labelled to all the sealer groups. All the sealers groups were manipulated according to manufacturer instructions and were coated on to the teeth using lentulospirals. Later all the samples were obturated using cold lateral compaction technique. The teeth sealed with intermediate restorative material (PREVEST DenPro) at the coronal end.

2.1 Sample preparation for confocal microscope

All the samples were sectioned orthogonally using double-sided diamond disk under continuous water cooling and obtained with a thickness of 1mm.

2.2 Evaluation of sample by using a confocal laser microscope

All the samples which sectioned at coronal, middle, and apical thirds examined with a Zeiss Pascal Laser Scanning Microscope (Carl Zeiss, Gottingen, Germany). Measurements were recorded using the digital measuring ruler, in CLSM image recorder software. The data were averaged to obtain a single value for each section. All analyses were recorded and evaluated by a single operator to rule out any discrepancies.

The data were subjected to statistical analysis using the statistical package for the social sciences IBM SPSS Statistics version 22.0 software and Oneway Analysis of Variance test for intragroup examination and Tukey's posthoc test for intergroup examination.

3. Results

The mean and standard deviation of penetration depth (mm) of the five sealers in the three different zones;

coronal, middle, and apical, are given in table 1. Among the sealers tested, Endosequence exhibited the maximum penetration at coronal, middle, and apical levels (Figures 1-3), whereas Endomethasone showed the least penetrability into the dentinal tubules. One-way ANOVA showed a significant difference (p=0.000) in the depth of penetration in the coronal and apical thirds in all the sealers. Maximum depth of penetration was observed at the coronal third, which was significantly higher than the depth of penetration observed at the middle and apical thirds for all five sealers tested.

In posthoc analysis, both Endosequence and MTA-Fillapex showed significant differences (p<005) with the other sealant materials in the coronal region (Tabe 2). However, Roekoseal exhibited no significant differences with AH-Plus and Endomethasone. In the middle zone, significant differences (p<0.05) observed between all the sealant materials (Table 3). In the apical zone, both Endosequence and MTA-Fillapex displayed significant differences (p=0.000) with all the sealants. Significant differences were also observed between AH-Plus and Endomethasone, whereas no significant differences were observed between Roekoseal and Endomethasone (Table 4).

4. Discussion

Factors influencing sealer depth penetration in dentinal tubules are the presence/absence of smear layer, dentinal permeability (the number and the diameter of tubules), root canal dimension, presence of water, and physical and chemical properties of the sealer [5]. In the present study, removal of the smear layer was done using 3ml of 17% ethylenediaminetetraacetic acid (DESmear, Anabond Stedman pharma research, India), which enhances the sealer penetration into the dentinal tubules.

Confocal laser scanning microscope (CLSM) images are distinctly higher than those obtained with the conventional optical microscope because the produced images contain volumetric and texture details that are impossible to achieve with the conventional microscope. The advantage of using CLSM is its higher resolution, greater contrast, three dimensions of reconstruction, image analysis. Hence in the present study, CLSM was used to measure the sealer penetration.

Results in the present study showed that in all the radicular portions Endosequence BC sealer showed the highest amount of penetration into the dentinal tubules followed by MTA Fillapex, AH-plus, Roekoseal and Endomethasone. The higher penetration of the Endosequence BC root canal sealer can be attributed to its extremely small particle size (less than 2 µm). Also, its low initial viscosity level and hydrophilic nature allow it to flow into all aspects of the canal anatomy. These specifications may improve the flow of the sealer into dentinal tubules, anatomic irregularities, and gutta-percha [6,7]. Moreover, Endosequence BC exhibits minimal or no shrinkage during the setting phase [8].

Table 1: Comparison of Surface roughness using One-way ANOVA

Sealers	Coronal third		Middle third		Apical third	
	Mean±SD#	Significance	Mean±SD	Significance	Mean±SD	Significance
Endosequence	1399.46±88.99	0.000*	1105.01±67.44	0.000*	591.89±66.52	0.000*
MTA Fillapex	1119.68±99.40		820.64±3.60		353.32±53.01	
AH-Plus	978.89±44.29		725.89±3.15		264.60±58.00	
Roekoseal	951.81±170.06		609.24±53.81		249.67±55.02	
Endomethasone	853.85±118.03		453.98±118.12		196.46±28.67	

* Significant differences were observed among the groups.

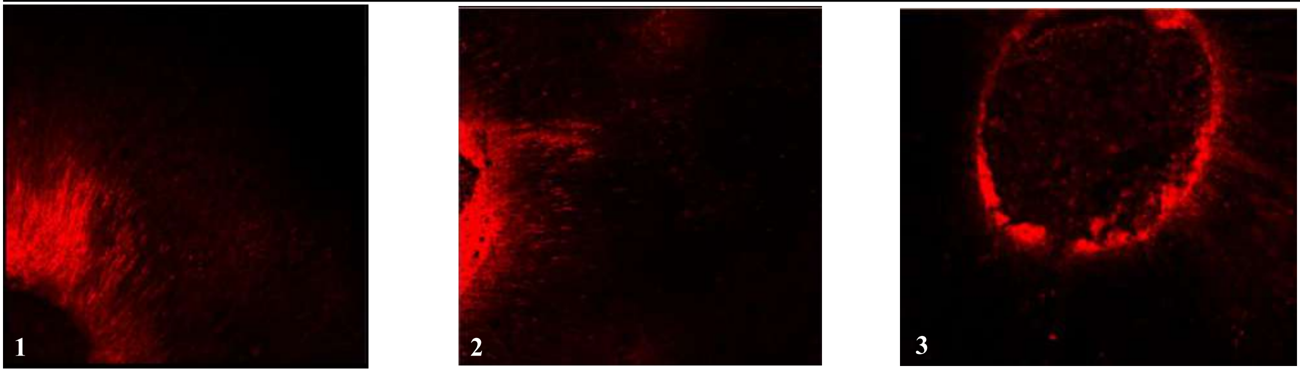

Figure 1-3: Conofocal Laser Microscope analysis of depth of penetration of Endosequence BC sealer. Where 1. at coronal level, 2. at middle level, and 3. at apical level .

Table 2: Intergroup Comparison of Depth of Penetration (μm) of the Sealers in the Coronal Zone.

Sealer group at coronal third	Groups	Mean Difference	Standard Error	Significance
	MTA-Fillapex	279.78658	45.68794	0.000*
Endosequence	AH-Plus	420.57308	45.68794	0.000*
	Roekoseal	447.64967	45.68794	0.000*
	Endomethasone	545.61067	45.68794	0.000*
	AH-Plus	140.78650	45.68794	0.026*
MTA-Fillapex	Roekoseal	167.86308	45.68794	0.005*
	Endomethasone	265.82408	45.68794	0.000*
AH-Plus	Roekoseal	27.07658	45.68794	0.976
	Endomethasone	125.03758	45.68794	0.061
Roekoseal	Endomethasone	97.96100	45.68794	0.217

* Significant differences were observed between the groups.

Table 3: Intergroup Comparison of Depth of Penetration (μm) of the Sealers in the Middle Zone.

Sealer group at middle third	Groups	Mean Difference	Standard Error	Significance
	MTA-Fillapex	284.36708	26.72156	0.000*
Endosequence	AH-Plus	379.11375	26.72156	0.000*
	Roekoseal	495.76958	26.72156	0.000*
	Endomethasone	651.02842	26.72156	0.000*
	AH-Plus	94.74667	26.72156	0.007*
MTA-Fillapex	Roekoseal	211.40250	26.72156	0.000*
	Endomethasone	366.66133	26.72156	0.000*
AH-Plus	Roekoseal	116.65583	26.72156	0.001*
	Endomethasone	271.91467	26.72156	0.000*
Roekoseal	Endomethasone	155.25883	26.72156	0.000*

* Significant differences were observed between the groups.

Table 4: Intergroup Comparison of Depth of Penetration (μm) of the Sealers in the Apical Zone.

Sealer group at coronal third	Groups	Mean Difference	Standard Error	Significance
Endosequence	MTA-Fillapex	238.57183	21.94659	0.000*
	AH-Plus	327.29342	21.94659	0.000*
	Roekoseal	342.22183	21.94659	0.000*
	Endomethasone	395.43150	21.94659	0.000*
MTA-Fillapex	AH-Plus	88.72158	21.94659	0.002*
	Roekoseal	103.65000	21.94659	0.000*
	Endomethasone	156.85967	21.94659	0.000*
AH-Plus	Roekoseal	14.92842	21.94659	0.960
	Endomethasone	68.13808	21.94659	0.024*
Roekoseal	Endomethasone	53.20967	21.94659	0.124

* Significant differences were observed between the groups.

In addition, the Endosequence BC root canal sealer exhibits a 0.2% expansion during the setting period. These characteristics also support the spread of sealer over the dentin walls of the root canal and filling of the lateral canals. All these features may contribute to the higher dentinal tubule penetration observed in the present study. This is in accordance with the literature reporting that tricalcium silicate–containing sealers penetrated into the tubules as deep as 2 mm due to the smaller particle size of BC Sealer [9] and also due to its high level of viscosity [10].

Penetration of MTA Fillapex is less when compared to Endosequence BC sealer as MTA Fillapex, a resin-based sealer has less than 20% MTA particles, and resin matrix shrinks 0.7% during setting. In contrast, the BC Sealer expands slightly (<0.1%), which may provide superiority for the latter [9,11]. However, MTA Fillapex has greater dentinal penetration than AH-Plus, Roeko Seal and Endomethasone. This greater penetration could be because of the presence of nano-particles, which enables a homogeneous mixture and a better flow of the sealer. MTA Fillapex is significantly more flowable, and this is attributed to the difference in composition and smaller particle size of the sealer [11-13].

The tubule penetration of resin-based sealers is not dependent on the hydraulic forces created during filling; instead, the sealer is drawn into the tubules by capillary action [1]. This may explain why AH Plus and Roekoseal, both with a longer setting time, exhibited significantly deeper penetration than Endomethasone. Endomethasone has the least penetration among all the sealers. Endomethasone contains both eugenol and paraformaldehyde, such as Endomethasone and N2, which were found to be the most toxic. Brodin et al. reported that Endomethasone could irreversibly inhibit the conduction of the action potential in the rat phrenic nerve [14].

In the present study, all the sealers exhibited the maximum penetration at the coronal third, followed by the middle third and least in the apical third. Various authors have demonstrated regional variation in the depth of tubular penetration [15-18]. Limitations of the present study include, temperature and humidity of the oral cavity are not simulated. Hence further ex-vivo and in vivo studies are needed.

5. Conclusion

From the present study, it can be concluded that the Endosequence BC sealer resulted in better penetration into the dentinal tubules. The maximum penetration of the five sealers was more in the coronal third followed by the middle third and least in the apical third.

Conflicts of interest: Authors declared no conflicts of interest.

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Comparison of remineralizing effect of organic and inorganic fluoride by evaluation of microhardness and quantitative analysis of calcium and phosphorus ratio on enamel surface: an *in-vitro* study

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K E Y W O R D S

Amine Fluoride

Sodium Fluoride

Microhardness

Enamel

Demineralization

Remineralization

SEM-Energy Dispersive X-ray analysis

A B S T R A C T

Background: Enamel is a highly mineralized tissue of the body which is composed of 96% inorganic salts and 4% organic matter. Enamel is permeable to water and ions, particularly cations and low molecular weight substances. The enamel continues to mature even after eruption with mineral replacing protein. Recently, the interest on the development of calcium phosphate-based remineralization technology has been increased that led to the development of various remineralizing agents like Fluoride, CPP-ACP (Tooth Mouse plus), Bioglass (Novamin), Ozone, Xylitol, Sensistat etc.

Aim: Aim of the present *in vitro* study was to evaluate and compare the remineralizing effect of Organic fluoride (AmF) and inorganic fluoride (NaF) by evaluating Vickers microhardness and quantitative analysis of Calcium and Phosphorus ratio on enamel surface using Scanning Electron Microscope-Energy Dispersive X-ray analysis.

Materials and methods: Sixteen maxillary central incisors were decoronated at the cement-enamel junction and mounted in cylindrical moulds filled with self-cure acrylic resin. Artificial demineralized lesions were created on the enamel surface by suspending them in 0.1 M Citric acid buffer at pH of 3.2 for 72 hrs. The samples were then randomly divided into two groups and labelled, Group A – remineralized with NaF for 3 minutes twice daily for one week and Group B - remineralized with AmF for 3 minutes twice daily for one week. Microhardness & SEM-EDX analysis were done before demineralization, after demineralization and after remineralization.

Results: Data were analyzed by comparing the mean values between the groups using independent sample t-test. The intra-group analysis was done using repeated-measures ANOVA with posthoc Bonferroni test, and a p-value of <0.05 was considered statistically significant.

Conclusion: Organic Fluoride resulted in better remineralization than inorganic Fluoride. After remineralization for one week, enamel samples treated with AmF demonstrated a statistically significant increase in mean microhardness and Ca:P ratio when compared to enamel samples treated with NaF.

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1. Introduction

Enamel is a highly mineralized tissue of the body, which is composed of 96% inorganic salts and 4% organic matter [1]. Enamel is permeable to water and ions, particularly cations and low molecular weight substances. The enamel continues to mature even after eruption with mineral replacing protein [2].

For many years, dental caries was considered as a progressive demineralization of enamel apatite followed by degradation of dentin. However, the present concept identifies caries as a dynamic process which can be conceptualized as an imbalance between mineral loss called demineralization and mineral gain called remineralization. Ultimately the net loss of mineral determines the progressive nature of caries.

The various causes of demineralization are acid dissolution of tooth mineral by plaque bacteria, other acidic sources (like carbonated soft drinks, citrus fruit juices, gastric reflux or regurgitation), decreased salivary flow [3] and sometimes intentional demineralization for micromechanical bonding of adhesive restorative materials. If the demineralization phase continues for a longer period, excessive loss of minerals results leading to loss of enamel structure and cavitation – the typical characteristics of dentinal caries.

During the past few years, there has been increased interest and development in calcium phosphate-based remineralization technology [4]. It is enhanced by providing low levels of Calcium and Phosphorus in conjugation with minimal amounts of Fluoride. A variety of remineralizing agents like Fluoride, Casein phosphopeptide- Amorphous calcium phosphate (CPP -ACP) (Tooth Mouse plus), Bioglass (Novamin), Ozone, Xylitol, Sensistatetc, that aid in remineralization of tooth structure are available commercially.

Fluoride is considered as the cornerstone of modern non-invasive dental caries management. Anticaries action of fluoride is due to formation fluorapatite, which is more acid-resistant than hydroxyapatite; enhances remineralization; inhibits ionic bonding during pellicle and plaque formation. In addition, fluoride also has an antibacterial effect [5].

Different types of fluorides used in dentistry are; Sodium fluoride (NaF), Sodium mono-fluorophosphate, Stannous fluoride & Acidulated phosphate fluoride (APF). These are inorganic fluorides and are available as varnishes, solutions, foams, gels, dentifrices etc. [6]. The important factor, which can effectively inhibit the caries is the bioavailability of fluoride. This availability of fluoride depends on its rate of solubility and the capability to adhere to the enamel [7]. In 1957, Muhleman *et al.* reported that organic fluoride (amino fluoride compounds) inhibits caries better than inorganic fluorides [8].

AmF is an organic compound such as N-octa decyl trimethylenediamine-N,N,N-tris(2-ethanol)-dihydro fluoride [$C_{27}H_{58}N_2O_{32}HF$], which consists of two functional groups such as a cationic amino organic group and abounds ionic fluoride group [9]. Recently, AmF containing dentifrices and mouth rinses are commercially available. However, limited research is available on comparison of the remineralization efficacy of various fluorides. Therefore, this study was designed to evaluate and compare the effect of organic fluoride (AmF) and inorganic fluoride (NaF) on calcium and phosphorus ratio on the enamel surface. The null hypothesis for this study was that there would be no difference between organic and inorganic fluoride in remineralization of enamel carious lesion.

2. Materials and methods

Sixteen permanent maxillary central incisors extracted for periodontal reasons were included in the study. The teeth were washed thoroughly and polished. The teeth were decoronated at cement-enamel junction using a high-speed diamond disc and mounted in acrylic resin. Subsequently, the mineral content and the microhardness were evaluated. The specimens were subjected to evaluate the mineral content (Ca/P ratio) and microhardness.

2.1 SEM EDX analysis

The mineral content (Calcium and Phosphorous) was evaluated using SEM-EDX (Zeiss Evo-18 model SEM, with EDX attachment of the Oxford model) prior to demineralization. The specimens were gold-sputtered and were subjected to scanning electron microscopy at 15.0 kV, and magnification of 10,000 X.

2.2 Vickers microhardness testing

The microhardness was evaluated using Vickers hardness tester ((UHL VMHT DIN 50 133). The specimens were placed on the Vickers hardness tester, and a load of 100g was applied for 15 seconds to produce the indentation. The indentations were made at three different points, and the mean of 3 measurements was recorded as baseline microhardness values.

2.3 Lesions creation on specimens

Artificial caries like lesions were created on specimens by suspending them in an artificial caries system, which is 0.1M citric acid buffer at pH of 3.2 for 72 hours [10]. After this procedure, the microhardness and mineral content of the specimens were again evaluated.

2.4 Treating the specimens with organic and inorganic fluorides

The samples were divided into two study groups (Figure 1) with eight specimens in each group. The experimental groups were distributed as follows:

Group A – Samples were treated with Sodium fluoride (NaF) for 3 min twice daily for one week.

Group B – Samples were treated with Amine fluoride (AmF) for 3 min twice daily for one week.

Specimens were again evaluated for their microhardness and mineral content after remineralization process for one week. In between treatment, the samples were stored in artificial saliva [10].

2.5 Statistical analysis

The obtained data were analyzed using statistical package for social sciences, SPSS 21.0, USA. The mean values were compared between the groups using independent sample t-test. The intra-group analysis was done using repeated-measures ANOVA with posthoc Bonferroni test, and a p-value of <0.05 was considered statistically significant.

3. Results

3.1 SEM-EDX Analysis

The SEM-EDX analysis (figures 2) revealed that after remineralization, calcium content increased significantly in the group treated with AmF when compared to NaF group. In contrast, there was no statistically significant difference in phosphate content in both groups. It also revealed that there was a statistically significant increase in fluoride level in AmF group when compared to NaF group (Table 1). ANOVA analysis showed significant differences in the calcium content ($p=0.009$), Ca:P ratio ($p=0.003$) and fluoride levels ($p=0.001$) after remineralization between the enamel samples treated with NaF and AmF (Table 1).

3.2 Vickers microhardness test

The enamel samples treated with AmF (Group B) demonstrated a statistically significant increase in mean microhardness when compared to enamel samples treated with NaF (Group A). Even though NaF group showed an increase in microhardness, but it is lower than baseline value which was statistically significant ($p=0.001$) while AmF group showed an increase in microhardness almost equal to the baseline value (Tables 2). However, no significant differences were observed among the specimens after demineralization.

4. Discussion

Fluorides are an important adjunct in the prevention of dental caries. Fluoride ions combine with hydroxyapatite crystals of enamel and forms fluorapatite

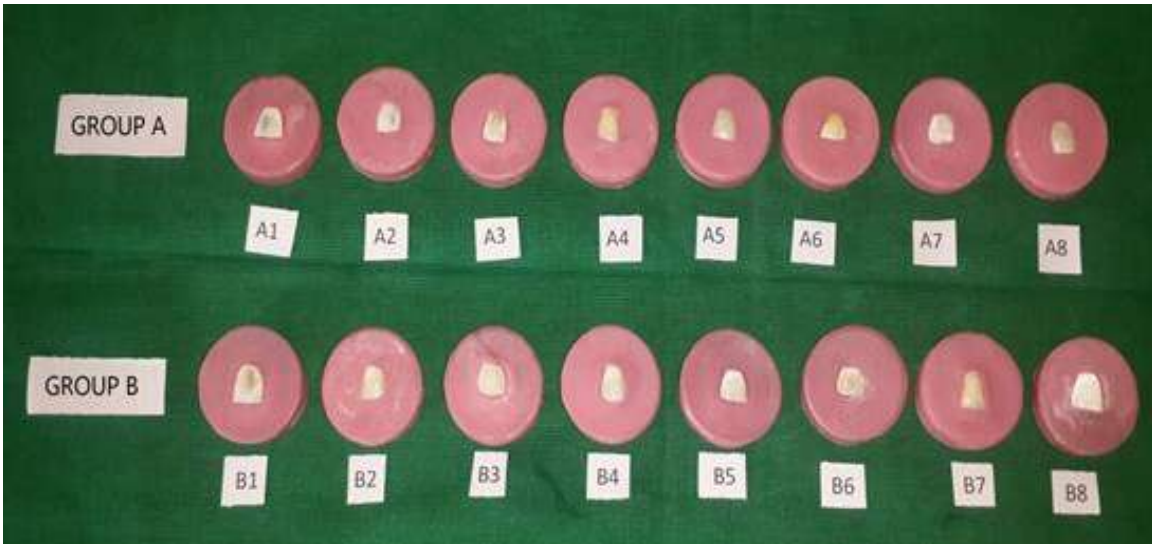

Figure 1: Decoronated maxillary central incisors mounted on acrylic resin .

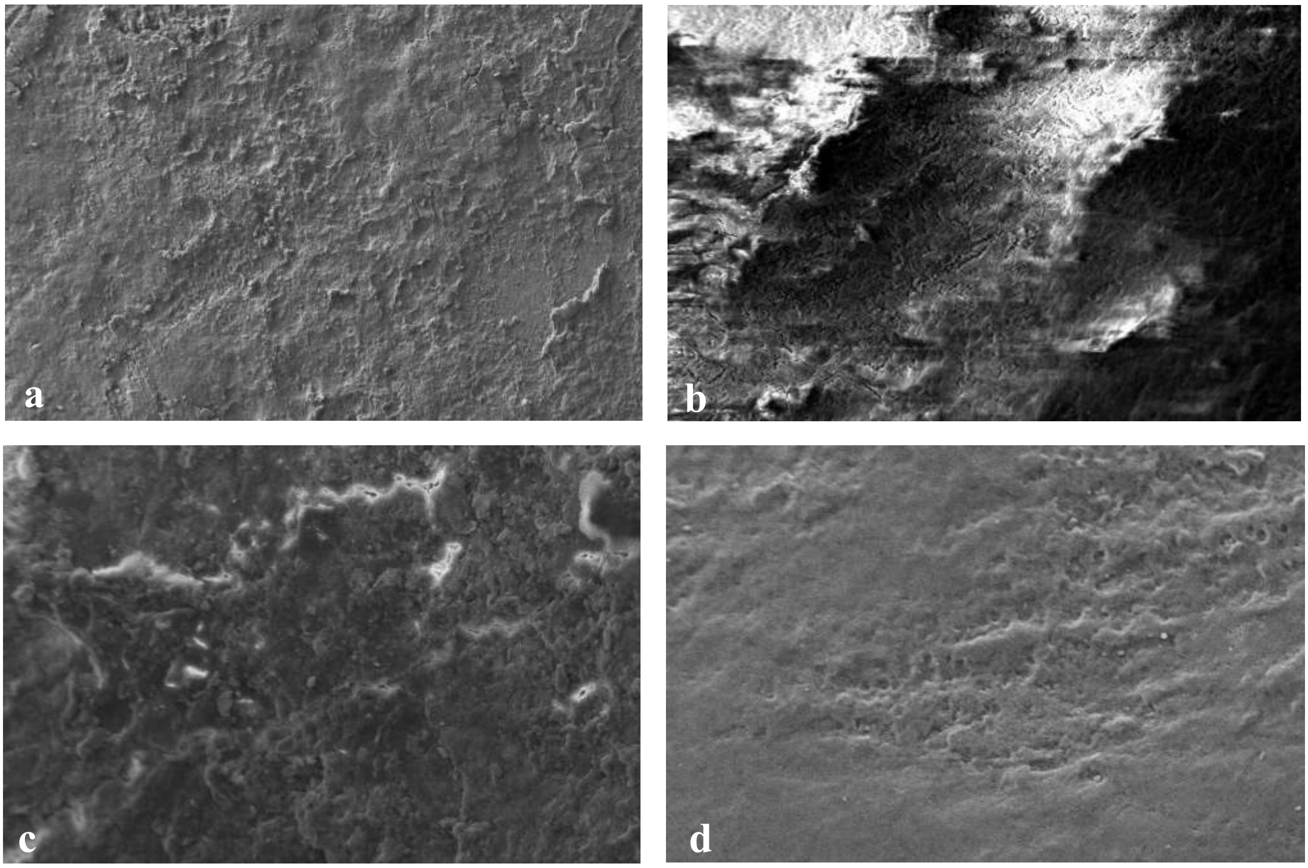

Figure 2: SEM-EDX analysis, where a. before demineralization, b. after demineralization for 72 hours , c. after remineralization for 1 week with sodium fluoride, and d. after remineralization for 1 week with amine fluoride

crystals, which enhance the remineralization of enamel [11]. Slow and sustained release of fluoride is necessary to have better deposition on the tooth surface and also to have its effectiveness over longer periods. Calcium ions combine with fluoride and forms the calcium fluoride (CaF_2), which results in slow release of fluoride and maintains the salivary fluoride level.

Dentifrices and mouth-rinses are the most commonly used topical agents. Numerous studies suggested that the use of fluoride mouth-rinses as they resulted in higher levels of oral fluoride retention than fluoride dentifrices [12]. Therefore, fluoride mouth-rinses were employed in this study.

Nozari *et al.* [13] reported that citric acid, lactic and acetic acids were all capable of demineralization and reduction of enamel micro hardness. In the present study, a solution of 0.1 M citric acid and 0.1 M Sodium citrate at pH 3.2 was used for demineralization of enamel samples [10]. The demineralization protocol was designed for 72 hours to simulate the duration that occurs in the oral cavity in caries susceptible individuals.

There are different methods such as direct and indirect techniques are available for evaluating demineralization and remineralization of enamel. Direct techniques include longitudinal microradiography, transverse microradiography and wavelength-independent X-ray microradiography. Indirect techniques are polarized light microscopy, microhardness measurement methods, Quantitative energy dispersive X-ray analysis, and iodide permeability.

The average microhardness value for human enamel was reported to be in the range of 270-370 KHN, the value in VHN range from 250-360 VHN which are very similar to each other. Also, the micro hardness (KHN and VHN) values are identical in dentin, where it is reported to be 50-70 KHN or 50-60VHN [14]. In the present study, Vickers hardness (VHN) was preferred over Knoop's hardness (KHN) because the square shape indent obtained in VHN is more accurate to measure.

Table 1. Inter-group comparison of EDX analysis of Ca, P, Ca: P and F content

		Groups				Significance
		NaF		AmF		
		Mean	SD	Mean	SD	
Ca	Baseline	68.64	3.23	67.64	2.42	0.497
	Demineralization	32.79	4.70	34.72	2.69	0.329
	Remineralization	34.45	3.95	39.55	2.67	0.009
P	Baseline	31.36	3.23	32.85	2.24	0.304
	Demineralization	17.65	2.33	17.99	1.02	0.711
	Remineralization	15.75	1.08	14.98	1.24	0.203
Ca:P	Baseline	2.20	0.21	2.06	0.10	0.112
	Demineralization	1.85	0.11	1.93	0.11	0.172
	Remineralization	2.18	0.20	2.61	0.27	0.003*
Fluoride		1.83	0.98	5.47	1.24	0.001*

* Significant differences were observed among the groups.

Table 2. Inter-group comparison of Vickers microhardness

		Groups				Significance
		NaF		AmF		
		Mean	SD	Mean	SD	
VHN	Baseline	380.88	23.65	387.88	25.26	0.576; NS
	Demineralization	302.25	11.25	307.75	21.90	0.538; NS
	Remineralization	342.13	21.38	385.25	20.57	0.001; Sig

* Significant differences were observed between the groups.

The baseline microhardness values obtained in the present study were in the range of 380.87-387.87 VHN. But, a decrease in the surface microhardness values for both the groups (302.25 and 307.75 VHN respectively) was observed after the demineralization process for 72 hours. After remineralization, the mean microhardness in Group A (NaF) increased to 342.12 VHN, whereas in Group B (AmF) it was 385.25 VHN (Table 2).

The results of the present study were in agreement with an investigation by Priyadarshini *et al.* [15]. They also suggested that AmF compounds result in a marked increase in enamel microhardness when compared to NaF. On the contrary, Lippert *et al.* [16] compared the anticaries potential of two new commercial dentifrices, which contain AmF and NaF; by measuring Vickers hardness and concluded that NaF showed superior anticaries potential when compared to AmF.

The EDX analysis of this study revealed that there was a statistically significant decrease in calcium and phosphate levels in both the groups after demineralization (figure 2.b). After remineralization, calcium content increased significantly in the group treated with AmF (figure 2.d) when compared to NaF group (figure 2.c).

In contrast, there was no statistically significant difference in phosphate content in both groups. It also revealed that there was a significant increase in fluoride level in AmF group when compared to NaF group (Table 1).

When enamel surface is treated with any fluoride dentifrice, the following reaction is anticipated.

$$Ca_{10}(PO_4)_6(OH)_2 + 20F^- \rightarrow 1OCaF_2 + 6P0_4 + 20H^-$$

$$\downarrow$$

$$Ca_{10}(PO_4)_6(F)_2$$

It is observed that calcium in hydroxyapatite remains in the salt as CaF2 while phosphorous content is released into the liquid as phosphate ions [17]. This reaction further progresses to form fluorapatite. This study revealed that Ca:P ratio increased in both the groups but the amount of increase is statistically more significant in the AmF group when compared to NaF group. This may be due to the advantageous characteristics of AmF, including its ability as a surface-active agent, which has tensioactive and anti-glycolytic properties. The surface-active property provides self-alignment of the hydrophilic part towards the tooth surface and the hydrophobic part towards the oral cavity that result in an accumulation of fluoride very close to the tooth surface. This accumulated fluoride readily combines with calcium and forms calcium fluoride, which acts as a fluoride reservoir [18].

The superior anti-cariogenic property of AmF can be explained by two reasons such as (a) Presence of fluoride, (b) the antiplaque effect of amine (organic) component that has the inhibiting effect on bacterial adhesion. Therefore, AmF allows accumulation of fluoride close to the tooth surface, providing a sustained fluoride release. Various studies have reported the AmF's anti-caries effects [19] based on their surface-active/tensioactive property that is leading to the fast distribution of fluoride and homogenous coating on the tooth surface for a prolonged period.

In the present study, treatment with NaF showed less remineralization on enamel surface. The reason for the less remineralization can be attributed to the formation of thick calcium fluoride layer on the tooth surface that might result due to the reaction between NaF (inorganic fluoride) and hydroxyapatite of enamel. This thick calcium fluoride layer inhibits further diffusion of fluoride from the topical fluoride agents, thus providing a relatively lower bioavailability of fluoride ions [20]. Further, the sodium cations do not have self-governing caries prophylactic property.

Arnold *et al.* [21] using polarized light microscopy had reported that the more stable superficial enamel layer was formed after treating with AmF compared to the treatments done with NaF or sodium monofluorides. In a study, Sefton J *et al.* [22] also suggested that the more amount of fluoride was deposited on enamel by treating it with AmF than sodium or stannous fluoride. Another study by Naumova *et al.* [23] used different amine concentrations on enamel remineralization. They concluded that the thickness of the superficial layer increased with decreasing fluoride concentrations, whereas Ca and P content increased with increasing fluoride concentration.

The results of this study suggested that use of NaF and AmF remineralizing agents results in remineralization of incipient lesions, thereby preventing further destruction of the tooth. Among the study groups, Group B (AmF) showed a more significant effect in remineralization when compared to Group A (NaF). Therefore, the null hypothesis for this study that organic and inorganic fluorides have a similar impact on remineralization of carious enamel lesion has been rejected.

5. Conclusion

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

- Both inorganic (NaF) and organic fluorides (AmF) were effective in remineralization. However, the enamel samples treated with AmF showed more Ca:P ratio compared to the treatment with NaF.
- Also, compared to NaF remineralization, AmF remineralization demonstrated a significant increase in mean microhardness, which is equivalent to the baseline values.

Further *in vivo* studies are to be undertaken to evaluate the efficacy of these remineralizing agents in remineralizing incipient lesions.

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A bibliometric analysis of the 100 most-cited articles in dental materials journals

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ABSTRACT

Background: Bibliometric studies are important as they provide an overview of research and scientific activity in a specific field. But studies of this type to determine the characteristics of the most cited articles in dental materials journals are completely lacking.

Aim: To analyze the characteristics of 100 most-cited articles in dental materials journals since its inception through April 2019.

Materials and methods: Google scholar database was used to retrieve the list of journals titled with the term "dental materials". A search was then conducted under "Publication Name" for each of the selected journals, and the articles were grouped by the category "Times Cited". The 100 highly cited papers published in five journals were contemplated in the eventual inquiry. The final collection was subjected to further scrutiny to determine the nature and characteristics of the documented revelations regard to journal name, year of publication, authors and their country, type of article, and area of research.

Results: The 100 most-cited articles were published between 1985 and 2016 with maximum publications during 2001-05. The number of citations was ranging from 1926to 304. Only Dental Materials (97), Dental Materials Journal (3), shared the list. The United States tops the list with 25 articles followed by Germany (12) and Belgium (11). Dental composites and adhesives were the most commonly addressed topics in dental materials journals.

Conclusion: This bibliometric analysis connoted the evolving and interesting research trends in dental material science.

1. Introduction

The field of dental materials has undergone more of a revolution than an evolution over the past 100 years [1]. Since then, it has acknowledged a good deal interest among clinicians and researchers by showing a shift from traditional silver amalgam to E-max restorations and from Ni-Cr-Co crowns to CAD-CAM zirconium crowns with a lot of innovations in dental materials [2]. The published

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literature in dental materials science is vast and, although easily accessible, clinicians, and researchers may not always assess the quality of publications that they read. Moreover, areas in which research has made dramatic progress may be difficult to identify, the challenge of identifying eminent research from among the multitude of journals and publications remains [3].

The citation index is broadly looked at as a necessary framework to adopt in the context of measuring relevance in scientific production [4,5]. The immensity and citations count received by an article does not embody the nature of its importance in the field of knowledge but cultivates and paves a stronger platform for exploring discourse in clinical practice, scientific assimilation, and furtherance of research in that particular field [5]. In this regard, bibliometric studies are of importance as they enable us to gain an overview and evaluate the intrinsic characteristics of published research in a particular field [6].

Numerous bibliometric studies have been conducted in different areas of material science. Kochhar examined the scientific literature from Indian institutions in various types of materials such as metals and alloys, ceramics, aluminum, glass, composites, polymers during 1980-83 [7]. The growth and size of the publication in the field of material science from 1993-2001 were analyzed by Walke and Dhawan [8]. Recently, a study was conducted to investigate the research and impact of materials science literature for the period of 1999-2008 [9]. However, no systematic investigations of this type have yet been published in the field of dental materials science.

As the practice of dentistry is defined by the current and future developments in the science of dental materials, the aim of this study was, therefore, to analyze the characteristics of 100 most-cited articles in dental materials journals.

2. Materials and methods

In April 2019, we conducted a search using Google Scholar Database to spot the most cited articles in dental material journals. In Google Scholar's metrics section, journals with titles containing the word "dental material" were searched from the publications catalogue. The following six journals were filtered:

i. Dental Materials
ii. Dental Materials Journal
iii. Journal of Dental Materials and Techniques
iv. The Journal of the Japanese Society for Dental Materials and Devices
v. Journal of Dental Biomaterials
vi. International Journal of Dental Materials

Only articles published in the English language were considered in the analysis. For this reason, the articles published in "The Journal of the Japanese Society for Dental Materials and Devices" were excluded as they were in the Japanese language. Then, all the articles published in the remaining five journals were grouped by category; "Times Cited" under "Publication Name". This provides a list of articles published in a given journal from the date of the first issue published until 30th April 2019, listed by citation count.

The final collection was assessed independently by two reviewers for the following characteristics: citation count, journal name, year of publication, authors affiliation, type of article (review articles, original articles, and systematic reviews/meta-analysis) and area of research (dental adhesives, dental cements, synthetic resins, dental alloys, dental ceramics, root canal filling materials, dental impression materials, dental implants, etc.) Only the first author/ corresponding author affiliation was considered in the analysis. The highest citation density parameter arranged the order of articles that have similar citation counts. A third reviewer's opinion was sought to obtain solidarity when there was disagreement. Descriptive statistics on the characteristics of the most cited articles were undertaken. Data analysis was performed using the Statistical Package for Social Sciences (SPSS, Version 18.0; IBM, Armonk, NY).

3. Results

Table 1 shows the list of 100 most-cited articles in dental materials journals until April 2019. The number of citations ranges from 1926 to 304. The first seven articles of the ranking exceeded 1,000 citations, and each of the first 36 had more than 500 citations. The most-cited paper to date in dental materials science is on "Surface treatments of titanium dental implants for rapid osseointegration" published in "dental materials" in 2007 (Table 1).

The 100 most-cited articles were published in only two

Table 1: List of the100 most-cited articles in dental materials journals

S. No.	Article	Number of Citations
1	Le Guéhennec L, Soueidan A, Layrolle P, Amouriq Y. Surface treatments of titanium dental implants for rapid osseointegration. Dent mater. 2007 Jul 1;23(7):844-54.	1926
2	Denry I, Kelly JR. State of the art of zirconia for dental applications. Dent Mater. 2008 Mar 1; 24(3):299-307.	1585
3	Ferracane JL. Resin composite—state of the art. Dent Mater. 2011 Jan 1;27(1):29-38.	1238
4	Bollenl CM, Lambrechts P, Quirynen M. Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: a review of the literature. Dent Mater. 1997 Jul 1;13(4):258-69.	1175
5	Breschi L, Mazzoni A, Ruggeri A, Cadenaro M, Di Lenarda R, Dorigo ED. Dental adhesion review: aging and stability of the bonded interface. Dent Mater. 2008 Jan 1;24(1):90-101.	1145
6	Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R, Pashley DH. Relationship between surface area for adhesion and tensile bond strength—evaluation of a micro-tensile bond test. Dent Maters. 1994;10(4):236-40.	1094
7	Ferracane JL. Hygroscopic and hydrolytic effects in dental polymer networks. Dent Mater. 2006;22(3):211-22.	1014
8	Peumans M, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, Van Meerbeek B. Clinical effectiveness of contemporary adhesives: a systematic review of current clinical trials. Dent Mater. 2005 Sep 1;21(9):864-81.	951
9	Van Meerbeek B, Yoshihara K, Yoshida Y, Mine AJ, De Munck J, Van Landuyt KL. State of the art of self-etch adhesives. Dent Mater. 2011 Jan 1;27(1):17-28.	918
10	Kern M, Wegner SM. Bonding to zirconia ceramic: adhesion methods and their durability. Dent Mater. 1998 Jan 1;14(1):64-71.	877
11	Guazzato M, Albakry M, Ringer SP, Swain MV. Strength, fracture toughness and microstructure of a selection of all -ceramic materials. Part II. Zirconia-based dental ceramics. Dent Mater. 2004 Jun 1;20(5):449-56.	873
12	Tay FR, Pashley DH. Aggressiveness of contemporary self-etching systems: I: Depth of penetration beyond dentin smear layers. Dent Mater. 2001 Jul 1;17(4):296-308.	857
13	Kosmač T, Oblak C, Jevnikar P, Funduk N, Marion L. The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic. Dent Mater. 1999 Nov 1;15(6):426-33.	821
14	Ruyter IE, Nilner K, Möller B. Color stability of dental composite resin materials for crown and bridge veneers. Dent Mater. 1987 Oct 1;3(5):246-51.	799
15	Kelly JR, Denry I. Stabilized zirconia as a structural ceramic: an overview. Dent Mater. 2008 Mar 1;24(3):289-98.	791
16	Weinmann W, Thalacker C, Guggenberger R. Siloranes in dental composites. Dent Mater. 2005 Jan 1;21(1):68-74.	771
17	Labella R, Lambrechts P, Van Meerbeek B, Vanherle G. Polymerization shrinkage and elasticity of flowable composites and filled adhesives. Dent Mater. 1999 Mar 1;15(2):128-37.	758
18	De Munck J, Vargas M, Van Landuyt K, Hikita K, Lambrechts P, Van Meerbeek B. Bonding of an auto-adhesive luting material to enamel and dentin. Dent Mater. 2004 Dec 1;20(10):963-71.	729
19	Pashley DH, Sano H, Ciucchi B, Yoshiyama M, Carvalho RM. Adhesion testing of dentin bonding agents: a review. Dent Mater. 1995 Mar 1;11(2):117-25.	728
20	Pashley DH, Tay FR, Breschi L, Tjäderhane L, Carvalho RM, Carrilho M, Tezvergil-Mutluay A. State of the art etch-and-rinse adhesives. Dent Mater. 2011 Jan 1;27(1):1-6.	697
21	Pashley DH, Tay FR. Aggressiveness of contemporary self-etching adhesives: Part II: etching effects on unground enamel. Dent Mater. 2001 Sep 1;17(5):430-44.	696
22	Miyazaki T, Hotta Y, Kunii J, Kuriyama S, Tamaki Y. A review of dental CAD/CAM: current status and future perspectives from 20 years of experience. Dent Mater J. 2009;28(1):44-56.	689
23	Bouillaguet S, Troesch S, Wataha JC, Krejci I, Meyer JM, Pashley DH. Microtensile bond strength between adhesive cements and root canal dentin. Dent Mater. 2003 May 1;19(3):199-205.	674
24	Ferracane JL. Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins. Dent Mater. 1985 Feb 1;1(1):11-4.	665
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Table 1: Continued..,

S. No.	Article	Number of Citations
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journals. The journal with the most significant number of articles cited was "dental materials" with 97 articles, followed by "dental materials journal" with three articles. Three journals did not have an article from the top 100 (Table 2).

Among the list, 40 articles were published during 2001-05, followed by2006-2010 with 25 articles. The most popular articles were original articles (65) and review articles (30). The predominant area of research in dental materials science was dental composites (42), dental adhesives (30) followed by research on ceramics (19). The majority of the articles were published from the United States (25), Germany (12) followed by Belgium (11). In quantitative terms, the author, with most articles (irrespective of whether they were corresponding or co-authors), was Pashley DH (9 articles) followed by Lambrechts Pand Tay FR (8 articles each). A total of 54 authors from 19 countries have contributed to citation classics in dental material science (Table 3).

4. Discussion

To the best of our knowledge, this is the first of its kind to explore the 100 most-cited articles in dental material journals. A bibliometric study of the most widely cited publications helps the discipline to identify important advances. It also offers us a longitudinal view of the speciality's conceptual development and identifies the scientific indicators in a particular area that may be behind the journals, researchers, institutions, or nations [10]. The current list of 100 most cited articles is considered classic because each of them had earned over 300 citations [11]. The number was greater for articles in the field of endodontics, which counts between 554 and 87 citations [12]. It was also much higher than orthodontic research, which ranged from 545 to 89 [4]. The citations are on par with articles published in periodontics, which ranged from 2307 to 229 [13]. Hence, it may be said that the amount of research in dental material science is much greater than endodontics and orthodontics.

Table 2: Distribution of the 100 most-cited articles in dental material journals.

Sl. No	Name of the journal	No. of articles	Scimago Journal metrics	Country, publisher, and Year of origin
1	Dental Materials	97	H index: 123 SJR 2017: 2.11	Netherlands, Elsevier, 1985.
2	Dental Materials Journal	03	H index: 47 SJR 2017: 0.57	Japan, Japanese Society for Dental Materials and Devices, 1988.
3	Journal of Dental Materials and Techniques	00	-	Iran, Mashhad University of Medical Sciences, 2012.
4	Journal of Dental Biomaterials	00	-	Iran, Shiraz University of Medical Sciences, 2014.
5	International Journal of Dental Materials	00	-	India, International Journal of Dental Materials, 2019.

Table 3: Characteristics of the 100 most-cited articles in dental material journals

	Characteristics	Number of articles
Year of publication	1985-1990	05
	1991-1995	09
	1996-2000	11
	2001-2005	40
	2006-2010	25
	2011-2015	09
	2015- to date	01
Type of article	Review articles	30
	Original articles	65
	Systematic reviews	05
Area of research	Dental composites	42
	Dental adhesives	30
	Dental ceramics	19
	Dental cements	09
	Endodontic materials	08
	Dental alloys	06
	Dental synthetic resins	03
	Dental implants	02
	Other materials	03
Country	USA	25
	Germany	12
	Belgium	11
	Italy	09
	Netherlands	08
	Japan	06
	UK	05
	Switzerland	04
	Sweden, Brazil	03
	Denmark, Norway, France, Australia,China	02
	Turkey, Slovenia, Finland, Liechtenstein	01
Authors	Pashley DH	09
	Lambrechts P, Tay FR	08
	Ferracane JL,VanMeerbeek B	07
	De Munck J	06
	Van Landuyt K,Feilzer AJ, Davidson CL	05
	Peumans M	04

The present analysis witnessed that the "dental material" journal alone had a more significant share with a meager contribution from “dental material journal”. It was also noted that journals with a higher H index contributed to the top 100. This indicates the researcher’s propensity to cite articles published in strong metric journals. The year of publication of an article matters for reasons for an increasing number of citations over time [10]. Typically, it is not cited until one or two years after publication, reaches a limit after three or ten years, then decreases [14]. This accounts for a minimum number of articles in the top 100 from the year 2015 onwards.

The research in dental material science was centered on dental composites with 40 articles from the top 100. With the introduction of usage of hybrid composites for rehabilitation in the 1980s, there has been an increase in the number of citations for research on composite materials [15]. The revolution in adhesive dentistry during the 1980s and 90s led to the maximum citations for research on dental adhesives [16]. Research on ceramics had swiftly increased in the 1990s and is continuing till date with the introduction of new techniques. Hence from this analysis, it is understood that with the dawning of a new field of study in each decade, research on those recent advances was more cited in comparison to other fields.

Interestingly, only two articles were related to dental implants; however, the research on dental implants is high on the other end. This could be possible that the research on dental implants is being published more widely in other journals than in dental material journals. In recent years, the interest in systematic reviews, their production, and their publication, has been growing as they became foundational to evidence-based dental practice [17,18]. But only 5% of the top 100 articles are systematic reviews. This indicates a paucity and needs for systematic reviews and Meta-analysis in focused areas of dental material science.

Our geographic analysis concurs with previously published data on highly cited articles in health research. The most-cited dental material research is concentrated exclusively in North America and Western Europe. A similar trend has been observed in citation analysis of other dental fields like Endodontics [12], Periodontology [13], implantology [19], and orthodontics [20] as well as in other medical specialities [21-24]. This phenomenon can be explained in part by an accumulative geographical advantage, as citations come more frequently from institutions based in the same country [20]. Another possible cause is the movement of eminent scholars from various parts of the world to these regions [21]. These results reflect the disproportionate impact of the United States on dental material science due to eminent scholars and the financial support provided to them [25].

The present study has a few limitations, along with the inherent problems of citation analysis. Bibliometric analysis, as with any method chosen, does not cover the entirety of scientific production. This is considered to be an important limitation of the present study as our search strategy did not identify articles published in other journals. These articles could not be included in the analysis, as it will be impractical to isolate them from among the innumerable journals. Self-citations and negative citations need to be considered for a meritorious analysis. It is often a common trend to cite the articles that were already cited many times or based on a popularity scale without any understanding of their current relevance and applicability. Finally, the study results need to be dealt with caution as the citation rates may be biased towards top-ranked journals and well-known scholars and can't be used for comparisons. We, therefore, think, how highly cited papers can yield to less biased measures that can complement citation rates.

5. Conclusions

The present bibliometric study provided valuable insights into the characteristics of highly cited articles in dental material journals. The 100 most-cited articles are considered important as they can provide information on advances, areas of most intense research and the future objectives in the field of dental material science.

Conflicts of interest: Authors declared no conflicts of interest.

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Surface roughness of acrylic denture base resins polished with different abrasive agents: an *in-vitro* study

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INFORMATION

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Surface roughness

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ABSTRACT

Background: Excessive surface roughness on the denture base can adversely impact the oral health of the patient. Therefore, it is necessary to polish the denture before they are delivered to the patient. The abrasive and polishing agents should provide a smoother surface without affecting the physical and mechanical properties of denture bases.

Aim: This study aims to examine and understand the potential of different polishing materials on surface roughness of acrylic denture base resins.

Materials and methods: A total of 60 Heat-cure acrylic specimens (acrylic bars) were made and grouped into six groups. Control (no abrasive), Pumice (Micro-white), Eggshell powder, Seashell powder, Black sand powder, White sand powder are used as abrasive materials for polishing these specimens. These polished specimens were subjected to profilometer surface roughness analysis.

Results: The acrylic specimens polished with eggshell powder on acrylic specimens showed the least surface roughness followed by black sand, white sand, pumice and seashell powders. Tukey HSD showed significant differences (p=0.000) between unpolished and polished specimens.

Conclusion: Eggshell powder effectively reduced the surface roughness of denture base resin material. However, the surface roughness demonstrated by all the abrasive materials used was within the threshold limit (2 µm). Therefore, all the materials can be used as abrasives.

1. Introduction

Acrylic resin is most commonly used for the fabrication of bases of removable partial dentures, complete dentures, the tooth-supported or implant-retained overdentures etc. [1]. PMMA (polymethyl methacrylate resin) material has desirable properties of excellent aesthetics, low water sorption and solubility, relative lack of toxicity, ability to repair, and simple processing techniques [1-3].

Surface roughness is an essential factor, which affects dentures by the accumulation of bacterial plaque and stains, leading to adverse impacts on oral health and makes the denture wearing patients to face difficulty for oral hygiene maintenance [1,4,5]. So, the removable complete or partial prosthesis must be highly polished before inserting into the patient's oral cavity. Biofilm is the slimy layer

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of colonies of microorganisms on any surface; the dental plaque is also an example of that biofilm only. Surface roughness is the finely spaced irregularities present on any surface, which can enhance the biofilm formation. If the denture surfaces are rough enough, they become a nidus for plaque accumulation as well as increase the adherence of microorganisms such as Candida albicans, Streptococcus oralis etc. [4,6]. So, the successful dentures should possess well finished and polished smooth surfaces intraorally [1]. It is one of the factors which can satisfy the patients also.

Pumice is one of the commonest fine dental abrasive used in dentistry, especially for prosthesis polishing [7]. It is used as a polishing agent on harder materials depending upon its particle size [8]. The pumice slurry is ideal to use as it reduces the generation of heat. The production of temperature may cause warpage to the non-metallic materials, and also wear away the brush. The wet pumice slurry keeps the work well covered with pumice and not allows the denture to slip off from the hand by the motion of the brush [9]. The only disadvantage of pumice is not readily available with processing. Unprocessed is not so useful for polishing purpose.

On the other hand, naturally available abrasives are widely used for polishing purpose in industries. The natural abrasives include eggshell powder, seashell powder, black sand and white sand [7]. Eggshell and seashells consist of calcium carbonate, which has superior abrasive properties [10]. Black and white sands consist of silicates (alumino-silicates), calcite, aluminium oxides and traces of other minerals like magnesium which help to enhance finishing properties of acrylic surfaces [11]. However, no substantial literature is available on the effects of these natural abrasives on the surface roughness of acrylic dentures. Hence, this study was designed to evaluate the effect of natural abrasives on the surface roughness of denture bases in comparison to pumice.

2. Materials and methods

Materials used in this study were Heat cure acrylic denture base materials resin (DPI Heat Cure, India), and abrasive materials including Pumice (Micro white, Asian Chemicals, India), Eggshell powder, Seashell powder, Black sand powder, White sand powder (Sheshrikisaan , India) are used as abrasive materials for polishing.

2.1 Preparation and finishing of acrylic resin specimens

A standard metal die (ISO standard 1567) with dimensions 10mm x 6mm x 3mm, was used for fabrication of acrylic resin specimens. Elastomeric putty impressions were made of the die and wax patterns were fabricated with the modelling wax with dimensions (10 × 6 × 3 mm). Moulds for acrylic resin specimens were prepared by flasking with dental stone according to conventional procedures. After dewaxing, packing was done with PMMA (Polymethyl methacrylate) heat cure acrylic material and cured by following long-curing cycle. A total of 60 specimens were fabricated. Finishing procedure of all test specimens was done by subjecting them to trimming with acrylic and tungsten carbide burs (Waldent, Premium, India). After that, they were hand-finished progressively using finer grades of silicon carbide paper with decreasing order of grit (emery paper numbers 80, 100, 120 and 220μm) and mandrel in unilateral direction and ten strokes for 10 seconds.

2.2 Preparation of abrasive powders

2.2.1 Preparation of Eggshell and sea shell powders

The seashells were collected from the sea coast. The collected Eggshells and seashells were washed, and then boiled at 100^{0}C, and vacuum dried in the microwave oven for 2min at 25^{0}C and crushed to powder using a blender (Prestige 730 Watts, India) for 40 minutes. After that powder was sieved for fineness with 25μm sieve.

2.2.2 Black sand and white sand powders:

Black sand and white sand were brought commercially from the aquarium shop. The sand was directly grounded in the mortar with pestle and then powdered twice using a blender for 40 minutes in two steps. After that powder was sieved for fineness with 25μm sieve.

2.3 Procedure of polishing with abrasive powders

A total of sixty samples were fabricated and divided into six groups, which comprises ten specimens (n=10) for each abrasive powders. Among the six groups, one is the control group, and the other five are for the individual abrasive powders such as pumice, eggshell, seashell, white sand, and black sand respectively.

The slurry with each abrasive powder was made by mixing the abrasive powders with 2 ml of distilled water.

A polishing felt cone was fixed on a dental lathe unit (Unident, India), and the abrasive pastes were applied. The acrylic specimens were polished by passing them across the felt cone, which was rotating at a speed of 1425 RPM. The acrylic specimens were polished for 2 minutes.

2.4 Evaluation of Surface Roughness

The polished specimens were tested for surface roughness using a profilometer (SRG 4000, I ndia) after polishing with each abrasive material. The specimen surface was fixed on a flat surface in a position to the horizontal base of the profilometer. The stylus (profilometer`s needle) was moved across the surface of each specimen two times in two different directions for a distance of 1.7 millimetres according to the apparatus design. The data was collected from the screen part of the profilometer.

The data were subjected to Oneway ANOVA and TukeyHSD tests for statistical analyses using SPSS for Windows, Version 21.0., SPSS Inc.

3. Results

The mean surface roughness of acrylic specimens polished with various abrasive agents was detailed in table 1. Statistical analysis showed that the surface roughness (Ra) was influenced by using polishing procedures compared to unpolished samples. Among the abrasive materials used, seashell powder on acrylic specimens showed more surface roughness (1.2760±.40484), and eggshell powder material on acrylic specimens showed less surface roughness (0.9510±0.51692) (Figure 1). One-way ANOVA showed significant differences (p=0.001) in the surface roughness among the materials tested (Table 1).

Posthoc analysis showed significant differences (p=0.000) between unpolished and polished specimens (Table 2). However, no significant differences were observed among the modified groups (Table 2). Though the polished acrylic specimens showed different mean surface roughness values, they were not statistically significant.

4. Discussion

Denture prostheses, which are widely used in dentistry, are made of heat-activated acrylic resin. Polishing involves removing rough surfaces incrementally. This may affect the physical and mechanical properties of acrylic resin, such as surface hardness [12-14]. Dental appliances can be polished through either mechanical or chemical polishing methods. For the fabrication of removable denture prosthesis, mostly polishing is done by mechanical polishing techniques [1,15,16]. In mechanical polishing, the surfaces are abraded by mechanical action and progressively reduce notches until a smooth polished surface is attained. In the chemical polishing method, the polishing agent penetrates through the surface of the denture prosthesis that results in breaking of the secondary bonds between the polymer chains, and finally promotes the

Table 1: Comparison of Surface roughness using One-way ANOVA

Groups	N	Mean	Standard Deviation	F value	Significance
Unpolished	10	2.2800	0.67728	9.846	0.001*
Pumice	10	1.2130	0.43405		
Egg Shell	10	0.9510	0.51692		
Sea Shell	10	1.2760	0.40484		
White Sand	10	1.0980	0.52115		
Black Sand	10	1.0730	0.29978		

* Significant differences were observed among the groups.

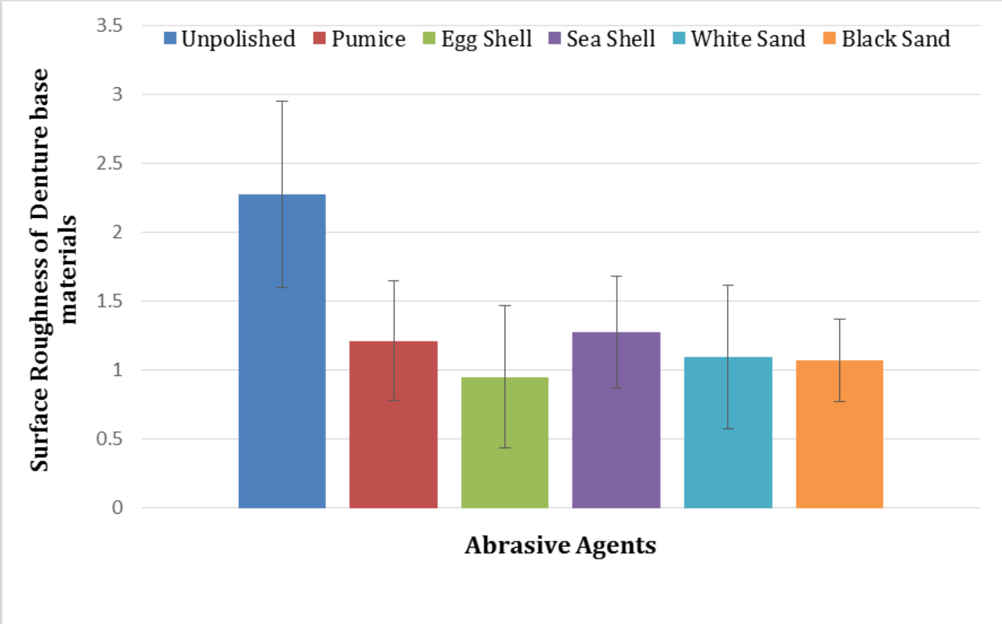

Figure 1: Surface roughness of denture base material polished with different abrasive agents.

Table 2: Pair-wise comparison of surface roughness using Posthoc analysis

Groups		**Mean Difference ± Standard Error**	**Significance**
Unpolished	Pumice	1.06700±0.21906	0.000*
	Egg Shell	1.32900±0.21906	0.000*
	Sea Shell	1.00400±0.21906	0.000*
	White Sand	1.18200±0.21906	0.000*
	Black Sand	1.20700±0.21906	0.000*
Pumice	Egg Shell	0.26200±0.21906	0.837
	Sea Shell	0.06300±0.21906	1.000
	White Sand	0.11500±0.21906	0.995
	Black Sand	0.14000±0.21906	0.987
Egg Shell	Sea Shell	0.32500±0.21906	0.676
	White Sand	0.14700±0.21906	0.984
	Black Sand	0.12200±0.21906	0.993
Sea Shell	White Sand	0.17800±0.21906	0.964
	Black Sand	0.20300±0.21906	0.938
White Sand	Black Sand	0.02500±0.21906	1.000

* Significant differences were observed between the groups.

plasticizing effect of the acrylic resin surface [17]. So, mechanical polishing was considered to be a better method, and it was the reason for choosing the mechanical polishing procedure in this study. Various studies also suggested that mechanical polishing produces significantly smoother surfaces on acrylic dentures compared to chemical polishing [17].

This present study evaluated the efficacy of different abrasive materials in comparison with pumice. Those abrasive materials are eggshell powder, seashell powder, black sand powder and white sand powder. These abrasive materials are used in the present study has their composition is almost similar and has properties same as pumice. The eggshell powder is composed of approximately 98.2, 0.9, 0.9% Calcium carbonate, Magnesium and Phosphorous (phosphate) respectively. Eggshell powder abrasive material consists of calcite, graphite tracers and thenardite [18,19]. They increase the abrasion rate and smoothness of dentures. The seashell powder contains calcium carbonate, silicon dioxide, aluminium oxide, which helps in an abrasive activity [9]. White and black sand consists of silicon dioxide, Aluminium oxide, ferrous oxide and Tracers of minerals which has abrasion properties [11].

Quirynen *et al.* [18] reported significant bacterial accumulation and their colonization would occur if the surface roughness is more than 2µm. Based on this study, the surface roughness of dental prostheses should not exceed 2µm. The plaque accumulation may result if the surface roughness is exceeded this threshold limit [20]. So, this threshold limit is considered as a basis to use an abrasive material to finish and polish the dentures.

In the present study, the eggshell material caused the least surface roughness compared to other abrasive materials. Whereas the specimens polished with seashell powders exhibited more surface roughness. However, the surface roughness of all the polished specimens with different abrasive powders was within the threshold limit (2 µm). Posthoc analysis showed significant differences between unpolished and polished specimens with different abrasive powder. However, no significant differences were observed between the polished specimens. This phenomenon indicates that all the five abrasive materials used in the study may be considered for polishing the acrylic denture prosthesis,

The results of this study were in agreement with Stanley *et al.*, who suggested that eggshell abrasive powder provides better smoother surfaces on the denture base acrylic resin than pumice [4]. The reason for this can be attributed to the hydrophilic nature of calcite ($CaCO_3$) and sodium sulphate coating present on the egg shells that aids to become instant slurry with water to enhance abrasive property [4,21].

Ahmed SA *et al.* [7] concluded that acrylic specimens polished with black sand exhibited higher surface roughness than white sand and pumice . Black sand possesses better mechanical and physical properties compared to white sand and pumice that made the black sand as a better abrasive material. Numerous SEM studies described the morphology of black sand powders that they contained fine and ultra-fine particles with an average particle size of 50 to 500 nm. This variation in their particle sizes made this material a better abrasive agent [22]. In contrast, black sand demonstrated less surface roughness compared to white sand in the present study.

Song E *et al*. [23] studied the effect of surface modification of $CaCO_3$, which is constituent in eggshell powder, by Laureth sulfonic acid surfactants on its wettability. They concluded that increase in surfactant concentration after the formation of a monolayer saturated with surfactant molecules produce a reverse change from hydrophobic to hydrophilic due to bilayer formation of surfactant molecules on the $CaCO_3$ surface [23,24]. The hydrophilic property of $CaCO_3$ makes it easy to become slurry with water to enhance dental polishability [25,26].

Al-Kheraif [27] reported a mean surface roughness (Ra) value of 0.10 mm on PMMA specimens polished with pumice; however, they used an automatic polishing machine, which is different from the conventional hand polishing method applied in the present study.

The eggshell powder showed less surface roughness than seashell powder, as the harder and finer the particles more will be the abrasive nature and properties of the polishing materials. Eggshell is most effective because of its composition and inclusion of LAS (Laureth sulfonic acid surfactants) coating, which increase the abrasion rate and causes smoothness of the dentures [20, 25].

5. Conclusion

From this study, the following conclusions were drawn;

- Unpolished group of acrylic specimens showed the highest surface roughness compared to polished acrylic specimen groups; so, it necessitates polishing the acrylic denture prosthesis before they delivered to the patient.

- Eggshell powder effectively reduced the surface roughness of denture base resin among all polishing materials used followed by black sand, white sand. However, the surface roughness demonstrated by all the abrasive materials was within the threshold limit (2 μm). Therefore, all the materials can be used as effective polishing agents in dentistry.

- Eggshells are available readily in every source, even domestically. So, its powder can be made abundantly at free of cost, unlike pumice powder, which is expensive and not readily available.

Conflicts of interest: Authors declared no conflicts of interest.

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Comparison of push-out bond strength of bioceramic sealer with bioceramic coated and non-bioceramic coated Gutta-percha: an *in vitro* study

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ABSTRACT

Background: The goal of the endodontic treatment is to achieve a fluid-tight impervious seal to prevent the ingress of bacteria and the occurrence of any pathology in future. The endodontic sealer plays a crucial role in obtaining the hermetic seal by filling all the spaces and by binding to the root dentin and to the core obturating material, which is usually gutta-percha.
Aim: This study aimed to compare the push-out bond strength of Endosequence BC sealer with bioceramic coated and non-bioceramic coated gutta-percha.
Materials and methods: A total of 36 extracted human maxillary central incisors were decoronated to standardize the root length of 15mm. Working length was determined and Biomechanical preparation for all the samples was done with a Mtwo (VDW Company) rotary file till 40/.06. Samples were randomly divided into three groups containing 12 teeth in each group based on the obturation procedure, namely Group 1: Endosequence BC sealer along with Endosequence bioceramic coated gutta-percha (n=12), Group 2: Endosequence BC sealer along with normal gutta-percha (n=12), Group 3: AH Plus sealer along with normal gutta-percha. All the samples were obturated using single cone technique. The specimens were sectioned orthogonally at middle third to obtain three sections of 1mm thick. All the samples were subjected to the push-out bond strength test with the universal testing machine. The data were subjected to one-way Analysis of Variance (ANOVA) followed by post hoc analysis.
Results: Samples obturated with Endosequence BC sealer with Endosequence bioceramic coated gutta-percha showed the maximum push-out bond strength followed by Group 3 and Group 2. One-way Anova showed significant differences (p=0.021) among the groups. In posthoc analysis, the specimens from group 1 exhibited significant differences (p=0.016) with the group 2 specimens.
Conclusion: The push-out bond strength of Endosequence Bioceramic sealer with Endosequence Bioceramic coated gutta-percha was significantly higher than that of Endosequence Bioceramic sealer with normal gutta-percha and AH Plus sealer with normal gutta-percha.

1. Introduction

The sound philosophy of endodontic therapy is to maintain a tooth in function in the dental arch following pulp pathology and sequelae, ideally in a way that is

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thorough and efficient. The success of endodontic therapy depends on thorough debridement of the root canal system, elimination of pathogenic organisms by establishing a fluid impervious seal. Gutta-percha is a commonly used obturating material. Gutta-percha does not bond to root dentin; hence, it is used in conjunction with a root canal sealer [1]. Traditionally, Zinc oxide Eugenol (ZoE) based materials are used as root canal sealers. However, they have some inherent drawbacks, including their inability to strengthen the root, as it does not adhere to dentin, microleakage, and the solubility of sealer. These shortcomings of ZoE sealants make their prognosis dilemmatic and unassured [2,3]. Hence, newer endodontic sealers are constantly being developed to provide enhanced properties.

AH Plus is an epoxy-resin based sealer, that is considered as the gold standard because of its physical properties [2]. The ability of this sealer to flow and its long-term polymerization time makes this sealer to penetrate deeper into the dentinal tubules and form strong mechanical interlocking between dentin and sealer.

Endosequence BC sealer is a recently introduced sealer, composed of zirconium oxide, calcium silicates, calcium phosphate monobasic, calcium hydroxide, and various filling and thickening agents. It is available in a premixed calibrated syringe with intracanal tips. As a hydrophilic sealer, it utilizes moisture within the canal to complete the setting reaction, and it does not shrink on setting [4]. Endosequence BC gutta-percha points are the latest innovative materials introduced into the field of dentistry [5]. Unlike the traditional GP points, these are subjected to a patented process of impregnating and coating each cone with bioceramic nanoparticles; they bond with the bioceramic particles in BC sealer to form a true gap-free seal. However, there is no adequate literature available on the bond strength of these sealant materials with the obturating materials. Therefore, this in vitro study was designed to compare the push-out bond strength of Endosequence BC sealer with bioceramic coated gutta-percha and non-bioceramic coated gutta-percha using the universal testing machine.

2. Materials and methods

A total of 36 specimens, which comprises 12 specimens in each group were used in this study, and it was confirmed using the G power software at a 95% confidence interval.

2.1 Preparation and obturation of root canals

A total of 36 extracted human maxillary central incisors were collected from the Department of Oral and Maxillofacial Surgery, Vishnu Dental College, Bhimavaram, Andhra Pradesh, India. The maxillary central incisors with a single root and single canal were included in the study. The teeth with calcified canals, cracks or fractures, development defects, multiple canals, root caries, and endodontically treated teeth were excluded. For standardization, all the samples were decoronated to a length of 15mm by using a double-faced diamond disc (KG Sorensen, Barueri, SP, Brazil). Pulpal tissue extirpation was done, and the working length was determined. Biomechanical preparation for all the samples was done in crown-down motion using Mtwo rotary nickel-titanium files (VDW, Munich, Germany) till 40/.06 size. Canals were irrigated between the use of files with 5ml of 3% Sodium hypochlorite (Prime dental PVT LTD., India). All canals were irrigated with 3ml of 17% ethylene di amine tetra acetic acid (DESmear, Anabond Stedman pharma research, India) to remove the smear layer. Final rinse performed by using 5 mL of distilled water to remove any remaining irrigating solution. All the irrigation procedure was followed using a side vented needle placed 1mm short of the apical foramen. The canals were dried with sterile absorbent paper points (Prime Dental PVT LTD., India.) after irrigation. All intracanal procedures were done by a single operator to eliminate inter-operator variability.

Teeth were then randomly divided into three experimental groups using computer-generated sequence allocation, consisting of 12 teeth each (n=12) based on the obturation procedure.

In Group 1, EndoSequence BC sealer (Brasseler, Savannah, GA, USA) syringe was inserted into coronal one third and gently dispensed a small amount of sealer into the canal by compressing the plunger of the syringe. Then by using a 15 hand file canal walls are lightly coated with the existing sealer in the canal. All the samples were obturated using single cone obturation technique with ISO number 40/.06 Endosequence BC master cone GP coated with sealer.

The procedure was the same in Group 2 as that of Group 1, but normal 40/.06 master cone GP was used for obturation instead of bioceramic GP.

In Group 3, AH Plus (Dentsply, Maillefer, Ballaigues, Switzerland) sealer is manipulated according to the manufacturer's instructions and is coated to canal walls using lentulospiral. Then all the samples were obturated using single cone obturation technique with ISO number 40/0.06 normal master cone GP. All the samples were coronally sealed using GIC type II (GC, Tokyo, Japan) and stored in distilled water for seven days to ensure complete setting of the sealers.

2.2 Sample preparation for evaluation of bond strength

Three 1 mm thick horizontal sections were prepared with the double-sided diamond disc under Continuous water-cooling from the middle third of each sample from all the groups.

2.3 Evaluation of bond strength

A suitable plunger, with 0.8 mm diameter, was selected such that the plunger did not contact surrounding dentinal walls when it was placed on the centre of the core material. The specimen was mounted on a universal testing machine (INSTRON-8801, Norwood, MA, USA). The load was applied at a crosshead speed of 0.5 mm/min in an apico-coronal direction to avoid any interference caused by root canal taper. At the time of dislodgement, the strength was recorded in megapascals (MPa) for each specimen.

The obtained data were analyzed using the statistical package for the social sciences IBM SPSS Statistics version 22.0 software, USA, and One-way Analysis of Variance test followed by posthoc analysis were carried out.

3. Results

The mean push-out bond strength (MPa) and standard deviations (SD) of segments of all the three groups are given in table 1. Group 1 samples showed the maximum push-out bond strength, and Group 2 samples exhibited the least bond strength.. One-way ANOVA test exhibited significant differences (p=0.021) among the groups. Posthoc analysis showed significant differences (p=0.016) between group 1 and group 2 specimens (Table 2) whereas group 3 specimens did not show significant differences with both the group 1 and group 2 specimens.

4. Discussion

Adherence of sealer to the root canal walls and GP are desirable properties for good, long term results [6,7]. Some of the physico-mechanical properties of sealers are specified in international standards such as EN-ISO 6876:2001, but they exclude certain properties such as adhesion to the canal wall [8]. Hence in the present study, the universal testing machine was used to compare the push-out bond strength of Endosequence BC sealer with bioceramic coated gutta-percha and non-bioceramic coated gutta-percha.

In the present study, Endosequence BC sealant material along with Endosequence bioceramic coated gutta-percha (group 1) showed the maximum push-out bond strength compared to the other two groups. The reason for superior bond strength in group 1 specimens can be attributed to the bonding between Bioceramic particles found in BC sealer and the Bioceramic particles

Table 1: Mean push-out bond strength (MPa) and standard deviations of segments of all the 3 groups (One-way ANOVA).

Groups	Mean	Standard Deviation	Standard Error	Significance
1	3.8974	1.01620	0.16937	
2	3.1456	1.19057	0.19843	0 .021*
3	3.4286	1.10314	0.18386	

Table 2: Comparison of push-out bond strength (MPa) of segments (Posthoc analysis)

Groups		Mean Difference	Standard Error	Significance
1	2	0.73389	0.26059	0.016*
	3	0.45083	0.26059	0.199
2	3	0.28306	0.26059	0.525

* Significant differences were observed among the groups.

in BC points. This bonding forms a true gap-free seal single cohesive unit called the Monoblock, which is a primary Monoblock pattern [9]. This monoblock pattern improves the bond strength of the sealer with dentin.

The creation of primary Monoblock in the root canal system [9,10] reduces the stresses that occur inside the tooth structure. Due to BC Sealer's ability to penetrate dentinal tubules and interact with dentine moisture, optimum dimensional stability and the least amount of shrinkage was obtained [11]. Hydroxyapatite is co-precipitated within the calcium silicate hydrate phase in setting reaction to produce a composite-like structure, reinforcing the set cement [12]. Similar to this present study, Kouvas V *et al.* (1998) [13] also concluded that the innovative Bioceramic-based sealer (Endosequence) might have the potentiality to strengthen endodontically treated teeth to a level comparable to that of intact teeth.

In the present study, Group 3 showed better results than Group 2. The higher bond strength obtained with Group 3 compared to Group 2. It can be attributed to its ability to combine with the amino groups that are exposed in collagen and form strong covalent bonds between the resin and collagen upon the opening of the epoxide ring [10,14]. The low bond strength value of Group 3 compared to Group 1 can be attributed to the fact that NaOCl despite its disinfectant properties, being a deproteinizing agent, it can degenerate dentin by collagen dissolution, affecting the resin sealer penetration and hindering the formation of a consistent hybrid layer. Furthermore, NaOCl breaks down into sodium chloride and oxygen that interfere with resin sealer (AH Plus) polymerization, causing strong inhibition at the sealer-dentin interface and hence decreasing the bond strength [15]. The results are in accordance with a study in which the bond strength of two sealants was compared with respect to the presence or absence of the smear layer. The adhesion strength of the BC-Sealer was superior to that of the AH-Plus without smear layer [16].

Group 2 showed the least bond strength values compared to other groups. In group 3, even though NaOCl decreased the push-out bond strength values of AH Plus sealer, it had more bond strength value than the Group 2. The reason for this lower Push-out bond strength in group 2 can be attributed to the poor bonding efficacy of the Endosequence BC sealer with the normal gutta-percha cones [17]. The results are in accordance with various studies [18,19] in which AH Plus showed greater bond strength as it is less soluble and epoxy resin component of AH Plus after water sorption may also have increased its resistance against dislodgement.

5. Conclusion

From this study, it can be concluded that the push-out bond strength of Endosequence Bioceramic sealer with Endosequence Bioceramic coated gutta-percha was significantly higher than that of Endosequence Bioceramic sealer with normal gutta-percha and AH Plus sealer with normal gutta-percha.

However, further in-vitro and in-vivo studies with a larger sample and the teeth with more complex anatomies are required to validate its clinical efficacy and applications.

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