University of Mosul
College of Dentistry

The Influence of Chitosan Nanoparticles as Final Irrigation on Smear Layer Removal and Bond Strength of Different Endodontic Sealers

A Thesis Submitted
By

Fatima Shamil Mohammed Salih Hasan

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Fanar Turki Aljadwaa

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بسم الله الرحمن الرحيم

﴿وَعِندَهُۥ مَفَاتِحُ ٱلْغَيْبِ لَا يَعْلَمُهَآ إِلَّا هُوَ ۚ وَيَعْلَمُ مَا فِى ٱلْبَرِّ وَٱلْبَحْرِ ۚ وَمَا تَسْقُطُ مِن وَرَقَةٍ إِلَّا يَعْلَمُهَا وَلَا حَبَّةٍ فِى ظُلُمَٰتِ ٱلْأَرْضِ وَلَا رَطْبٍ وَلَا يَابِسٍ إِلَّا فِى كِتَٰبٍ مُّبِينٍ ﴿٥٩﴾﴾

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ABSTRACT

The aims: this in vitro study aims to evaluate the effect of chitosan nanoparticles irrigant with two concentrations (0.2% and 0.5%) on smear layer removal, bond strength and failure modes of AH Plus, MTA Fillapex, and EndoFill sealers to the radicular dentine and compared to other irrigatants.

Materials and methods: A total 108 human lower premolar teeth with single, straight-roots were obtained after being freshly extracted. The crown of all teeth was transect to leave a 12-mm root length. Each root was accessed and working length determination. Preparation of canals was carried out through Pro-Taper universal rotary system files up to the size F3. During instrumentation and between each file, the canal was irrigated with 2 ml of 2.5% NaOCl followed by 5ml of distilled water to flush it. Based on final irrigation six groups (n =18) and each final irrigant were rinsed the canals about 3 ml for 3 minutes. Group I: 0.2% Chitosan nanoparticles solution (CNPs), Group II: 0.5% Chitosan nanoparticles solution (CNPs), Group III: 17% by Ethylenediaminetetraacetic acid (EDTA), Group IV: 3% Sodium hypochlorite (NaOCl), Group V: 2% Chlorohexidine (CHX) and Group VI: Distilled water as a control group. From each group nine samples were taken for smear layer removal test and each sample was longitudinally sectioned and observed under scanning electron microscope (SEM) at 2000X magnification. Finally, the collected data was analyzed statistically. Other nine samples from each group were utilized for push out bond strength and distributed in 3 groups (n=3) based on endodontic sealer was used. Group A: AH Plus sealer, Group B: MTA Fillapex, Group C: EndoFill sealer. All sealers were used according to manufacture s instructions. The canal of each sample were obturated using single cone technique and incubated for 7 days at 37 C° and 100% humidity in incubator. From each sample, three slices, two mm thick, were cut from coronal, middle, and apical levels. A universal

testing machine was used to analyze push out bond strength (POBS) at a cross-head speed of 1mm/min, and a stereomicroscope with a 20X magnification was utilized to examine the mode of failure. Then data were collected and statistically analyzed using Kruskall-Wallis test, Mann-whitney test and Post-hoc test for smear layer removal test and Kruskall-Wallis test for push out bond strength test.

Results: The statistical significant difference was found among the groups in all three thirds ($p<0.01$) in Kruskall-Wallis test in comparison between 0.2% and 0.5% CNPs groups with other test groups and no significant difference between 0.2% and 0.5% CNPs ($p>0.05$) in Maan-Whitney test. CNPs were highly efficient as chelating solutions apically, but equally effective in coronal and middle thirds to 17% EDTA. The efficiency of 3% NaOCl at cervical and middle levels was superior to CNPs. 2% CHX and distilled water had the least efficient on removing the smear layer at all levels. Kruskal-Wallis test showed no statistically difference between irrigations on the effect of push out bond strength of three test sealers. The highest mean POBS at apical third is CNPs followed 17% EDTA then 3% NaOCl. 2% CHX increase POBS of AH Plus sealer and MTA Fillapex at coronal and middle respectively. The lowest mean POBS is 3% NaOCl and distilled water.

Conclusions: Newly irrigation CNPs appear to be high efficient in smear layer removal as final washing than 17% EDTA especially at apical third. Also CNPs improve bond strength of AH Plus, MTA Fillapex and EndoFill sealers better than 17% EDTA. 2% CHX is as effective as CNP in improve bond strength of resin based AH Plus, MTA Fillapex and EndoFill sealers while 3% NaOCl have adversely effect on POBS of sealers. All irrigations reduce the bond strength of EndoFill sealer except 0.2% and 0.5% CNPs.

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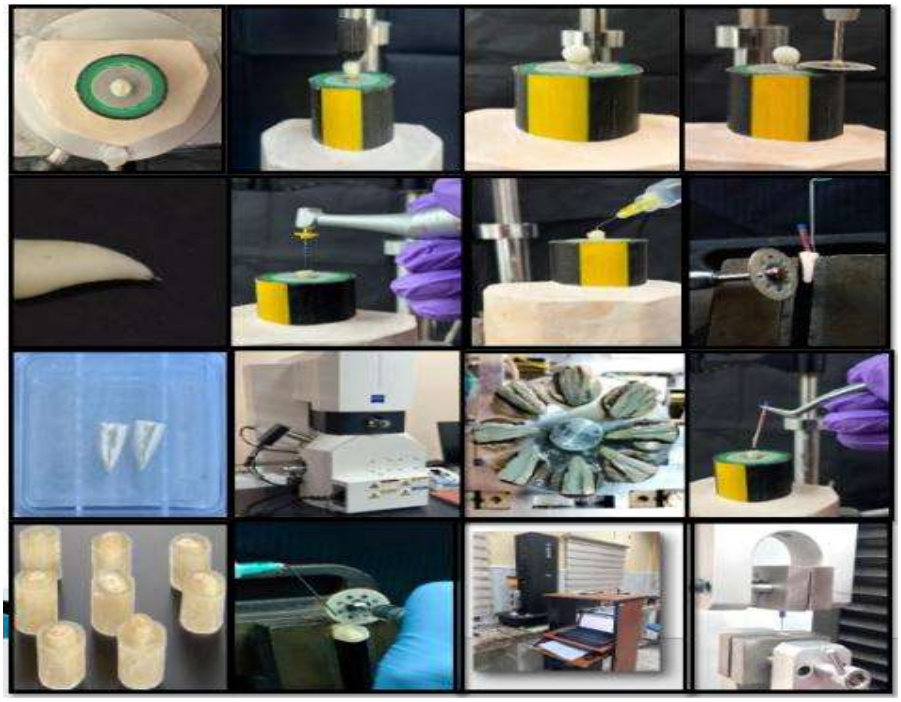
Author: Fatima Shamil Mohammed Salih Supervisor: Fanar Turki Abdulhameed Publisher: University of Mosul

HIGHLITIS

1. 0.2% and 0.5% CNPs irrigations are the more effective in eliminating smear layer than other tested irrigation solutions at apical level of root canal. They have a positive outcome on push out bond strength of all tested sealers.
2. 17% EDTA have a poor effect on push-out bond strength of all tested sealers.
3. 3% NaOCl is the most effective in eliminating smear layer among the tested irrigations at coronal and middle levels and have negative effect on push out bond strength of all sealers.
4. Distilled water have the least effect on smear layer removal at all levels and on the push-out bond strength of all tested sealers.
5. 2% CHX have the least effect on smear layer removal at all levels but improves the push-out bond strength of all sealers.
6. AH Plus sealer have a higher push-out bond strength than MTA Fillapex sealer while EndoFill sealer have the least bond strength at all levels of all irrigation solutions.

GRAFICAL ABSTRACT



Keywords:

Chitosan nanoparticles (CNPs)

Ethylenediaminetetraacetic (EDTA)

Scanning electron microscope (SEM)

Push out bond strength (POBS)

Failure modes

ABSTRACT

Aims: This in vitro study aims to evaluate the effect of chitosan nanoparticles irrigant with two concentrations (0.2% and 0.5%) on smear layer removal, bond strength and failure modes of AH Plus, MTA Fillapex, and EndoFill sealers to the radicular dentine. **Materials and methods**; A total 108 human lower premolar teeth. The crown of all teeth was decoronated to leave a 12-mm root length. Preparation of canals was carried out through Pro-Taper universal rotary system files. During instrumentation and between each file, the canal was irrigated with 2 ml of 2.5% NaOCl followed by 5ml of distilled water to flush it. Based on final irrigation six groups (n =18) and each final irrigant were rinsed the canals about 3 ml for 3 minutes. Group I: 0.2% CNPs, Group II: 0.5% CNPs, Group III: 17% EDTA, Group IV: 3% NaOCl, Group V: 2% CHX and Group VI: Distilled water as a control group. From each group nine samples were taken for smear layer removal test, sectioned longitudinally and observed under scanning electron microscope (SEM). Other nine samples from each group were utilized for push out bond strength and distributed in 3 groups (n=3) based on endodontic sealer was used. Group A: AH Plus sealer, Group B: MTA Fillapex, Group C: EndoFill sealer. The canal of each sample were obturated using single cone technique and incubated for 7 days at 37 C° and 100% humidity in incubator. From each sample, three slices, two mm thick, were cut from coronal, middle, and apical levels. A universal testing machine was used to analyze push out bond strength and stereomicroscope was utilized to examine the mode of failure. **Results:** The statistical significant difference was found among the groups in all three thirds ($p<0.01$) in Kruskall-Wallis test in comparison between 0.2% and 0.5% CNPs groups with other test groups and no significant difference between 0.2% and 0.5% CNPs ($p>0.05$) in Maan-Whitney test. Kruskal-Wallis test showed no statistically difference between irrigations on the effect of push out bond strength of three test sealers. **Conclusions** Overall, CNPs appear to be high efficient in smear layer removal as final washing especially at apical third. Also CNPs improve bond strength of all tested sealers better other irrigants. 2% CHX is as effective as CNP in improve bond strength of three tested sealers while 3% NaOCl have adversely effect on POBS of sealers. All irrigations reduce the bond strength of EndoFill sealer except 0.2% and 0.5% CNPs.

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INTRODUCTION

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INTRODUCTION

1.1 Introduction

It is commonly known that not all of the root canal's internal surfaces can be cleaned by mechanical preparation. The irrigation important in eliminating the smear layers and necrotic tissues. It also removes bacteria and their products from the canal (Wiaam, 2011; Ratih *et al*., 2020; AlEraky *et al*., 2023).

The Smear Layer is an uneven layer of denatured debris that forms on dentinal walls due to cutting action on the endodontic instrument. It is an amorphous structure that contains both organic and inorganic phases. It comprises microcrystalline, organic particle debris, dentine particles, bacteria, residual necrotic and vital pulp tissues, and odontoblastic processes. This prevents sealants, intracanal medications, and root canal irrigants from entering the dentinal tubles, and prevents the endodontic sealer from making intimate contact with the dentin walls (Rao *et al*., 2021).

Numerous endodontic irrigants have been developed. These include oxidizing agents like Hydrogen peroxide (H_2O_2), chelating agents (like EDTA, bisphosphonate HEBP, natural agents (like Triphala, chitosan, propolis), and antibacterial agents (like NaOCl, CHX, and MTAD). Although Qmix, Tetraclean, and Smear clear combinations have been used. 0.5% to 5.25% of NaOCl is a widely used irrigant during root canal preparation because of its antibacterial action and capacity to discard organic remnants and biofilm. A qs a result, it's unable to remove the smear layer a lonely. Therefore, it has been suggested that the smear layer is to be eradicated during endodontic treatment using a chelating agent like chitosan (CH) or EDTA (Kaushal *et al*., 2020).

The most popular method for getting rid of the smear layer's inorganic component is EDTA. It can eliminate the smear layer created during canal shaping. However, studies have shown that it has adverse effects on the radicular dentin underneath the smear layer as being calcium-depleting irrigant. Dentinal erosion may result from using 17% EDTA in root canals for a period longer than a minute. Moreover, EDTA may have demineralizing effects on dentin in root canals, which would lower dentin micro-hardness (Huang *et al*., 2023).

Root canal filling in endodontic treatments should provide a three-dimensional, fluid-tight seal of the prepared and disinfected space. The most often utilized material for obturation is gutta-percha (GP), however, because GP does not adhere to the root dentin, a sealant must be used in order to create an appropriate sealing at the interface. When employed in a lateral compaction approach, root canal sealers fill the area between the GP and the canal wall, flowing into lateral abnormalities, accessory canals, and spaces between GP points (Lim *et al*., 2020; Alkahtany *et al*., 2021).

The selection of root-filling materials is influenced by the ability of the sealer to penetrate effectively and consistently into dentinal tubules and their adaptation to the canal walls. It is thought that removing the smear layer from the root canal walls is essential to enabling sealer penetration (Arikatla *et al*., 2018).

To guarantee that the smear layer is completely removed, it must be properly identified. SEM is used to identify the smear layer and the opening of dentinal tubules (Mankeliya *et al*., 2021).

The push-out bond strength (POBS) test is a widely used method in dentistry to assess the bond strength between root canal filling materials and the dentin walls of a tooth. It is particularly valuable for evaluating the seal created by root canal sealers and for assessing the dislodgement resistance of root canal fillings. The test can also be used to assess the extent of sealer

penetration into the dentine tubules. POBS testing allows for the assessment of the bond strength of the root canal sealer at various levels along the canal, even in areas with poor adhesion (Ghouchani *et al*., 2021).

1.2 Aims of the study

The aims of this in vitro study are:

1. Evaluate the effectiveness of two different concentrations from chitosan nanoparticles solution (CNPs) and compare them to other golden standard solution in the removal of the smear layer at different level of the root canals using scanning electron microscope.
2. Evaluate the effect of chitosan nanoparticle solution (CNPs) with two different concentrations on bond strength and failure modes of three different sealers to radicular dentine and compare them to other irrigations at different levels of the root canals using push out test under universal testing machine.

1.3 Null hypotheses of the study

The null hypotheses of this study include the following:

1. The study hypothesis is that there would be no differences in Smear layer removal after the final rinsing by various irrigation solutions, which involved 0.2%, 0.5% CNPs, 3% NaOCl, 17% EDTA, and 2% CHX.
2. The null hypothesis is there would be no effect on the POBS of tested sealers after the final rinsing by various irrigation solutions, which involved 0.2%, 0.5% CNPs, 3% NaOCl, 17% EDTA, and 2% CHX.
3. There would be no difference between tested sealers in POBS.

CHAPTER TWO
LITERATURE REVIEW

CHAPTER TWO
LITERATURE REVIEW

2.1 Root Canal preparation

Root canal treatment (RCT) is the indicated endodontic therapy in cases of irreversible pulpitis and/or apical periodontitis. Chemo-mechanical root canal preparation involves both of mechanical instrumentation and antibacterial irrigation. Tools like engine-driven rotary files and hand files are needed to prepare root canals mechanically (El-Kishawi and Khalaf, 2021; León-López *et al*., 2022).

ProTaper Universal is one of the most widely utilized systems. Its system-based efficiency produces a remarkable performance in terms of precision and adaptability. Their convex triangular cross-section and progressive taper design are two key characteristics that increase cutting efficiency and flexibility while lowering torsional loads and file fatigue (Jordan *et al*., 2021; Roghanizad *et al*., 2017).

2.2 Smear Layer

In order to establish a bacteria-free canal, endodontic instruments must completely remove dentin and pulpal material from the root canal wall. However, as a result of their effect on the root canal walls, all endodontic devices produce dentin debris and a smear layer. The "muddy" from the "dusty" pattern of superficial debris is a result of endodontic instrument scratching and troweling, compacting an amorphous layer of organic and inorganic debris and occasionally bacteria up against the dentin walls (Dennis *et al*., 2021; Kiran *et al*., 2016).

A number of variables, such as the type and quantity of the irrigating solution, the size and shape of the root canal, the shape and sharpness of the tools, and the wet or dry cutting of the dentin, might affect the thickness of the smear layer from tooth to tooth (Al Shehadat, 2017).

The radicular smear layer acts as a barrier, partially obstructing and sealing the dentinal tubules (smear plugs), reducing dentin permeability by up to 86%, making it difficult for substances used as intracanal medication to diffuse, preventing the penetration of the endodontic sealers into the dentinal tubules (Özcan and Volpato, 2020). The existence of a smear layer is known to significantly reduce the cementing medium's tensile strength. It is also well known that the presence of a smear layer increases micro-leakage. However, several investigations disagree with the detrimental consequences of the smear layer. However, during endodontics, clinicians frequently employ a variety of irrigants to eliminate the smear layer. A canal filled without continuous irrigation will result in a thicker smear layer than a canal filled with continuous irrigation (Bhagwat *et al*., 2016). The number of cutting edges, their diameter and the rotatory files of the instruments affect the amount of smear generated. However, one study noticed no difference in theamount of smear layer between canals with different tapers. The use of coarse diamond burrs produce a thicker layer compared to carbide burrs. Hand instruments are also considered to produce severe smearing of the dentin due to application of high forces (Purohit and Purohit, 2021).

Debris is defined as dentin chips, pulp remnants, and particles loosely attached to the root canal wall. During cleaning and shaping, organic components of pulp tissue and inorganic dentinal debris accumulate on the radicular canal wall, producing an amorphous, irregular smear layer. Apical extrusion of debris is associated with symptomatic apical periodontitis (Nevares *et al*., 2017)

Accumulation of debris may prevent adequate sealing during root canal filling, and in cases with apical periodontitis reduces the chance of healing. The residual smear layer on the root canal walls may become disintegrated or removed by bacterial byproducts allowing leakage. However according to another concept, the smear layer may prevent bacteria from penetrating the dentin tubules and its contamination. It prevents detergents and sealers from

penetrating tubules. Based on a prior review the smear layer dramatically affects the micro leakage of apical and coronal areas and consequently the long-term success of the treatment. Therefore, this layer of organic and inorganic materials must be removed before filling the root canal (Ashraf *et al*., 2023).

2.3 Techniques for Delivering and Activating Irrigants During Endodontic Treatment (Raducka *et al*., 2023)

1. Manual passive irrigation (syringe\needle; brushes; manual dynamic agitation).
2. Machine assisted agitation (rotary brushes; Quantec-E irrigation system (SybronEndo, Orange, CA).
3. Thermal technique (extracanal heating of the solution before injection to the root canal) and intracanal (heating directly inside the root canal with heat carriers like System-B - Endodontic Heat Source, Kerr Endodontics, Gilbert, USA or alike
4. Passive Ultrasonic Irrigation (PUI).
5. Sonic techniques (EndoAcivator, Dentsply Maillefer, Ballaigues, Switzerland; EDDY, VDW, München, Germany).
6. Pressure techniques (EndoVac, Kerr Endodontics, Gilbert, AZ, USA; Rinsendo, Dürr Dental, Bietigheim, Germany).
7. Laser techniques.

2.4 Methods of Smear Layer removal

Three main methods are used to remove smear layer: Laser, Ultrasonic, Chemical or a combination of them.

2.4.1 Laser Removal

2.4.2 Ultrasonic Removal

2.4.3 Chemical Removal

Smear layer removal techniques involve copious irrigation using chemical solutions that dissolve its organic and inorganic components.

Irrigation materials are being routinely used for root canal treatment. As well as mechanical preparation to ensure successful root canal treatment (Abdelkafy *et al*., 2023; Abognah *et al*., 2020).

2.5 Irrigations

The two goals of root canal irrigation are the physical goal and the biological goal. The physical goal tries to achieve effective debridement by encouraging the irrigant to flow down the whole root canal while causing enough physical interaction with the root canal wall (Ali *et al*., 2022).

The biologic goal aims to remove the smear layer from the canal walls, dissolve tissue debris, breaking up bacterial biofilms, and destroying endotoxins. Several chemicals have been suggested for root canal irrigants. However, no single irrigant has all these desirable properties (Rath *et al*., 2020; Tartari *et al*. 2018; Zollinger *et al*., 2019).

Properities of irrigations (Gomes *et al*., 2023)

1. Low surface tension
2. Low viscosity
3. Broad antimicrobial activity
4. Effective on biofilm
5. Tissue solvent activity
6. Substantivity
7. Lubricant
8. Colorless and don’t stain the teeth
9. Stability in solution
10. Suspension of debris
11. Chelating activity
12. Biocompatible with periapical tissues
13. Gutta-percha disinfection
14. Little potential to cause an anaphylactic
15. Being active in the presence of blood, serum, and tissue protein derivates
16. They don’t interfere with the repair of periapical tissues.

17. They don’t interfere with the physical properities of the dentine (modulus of elasticity and flexural strength).
18. They don’t interfere with restorative material-dentine adhesion.
19. They don’t interfere with stem cell viability.
20. Being inactivate endotoxin or virulence factors.

2.6 Classification of Endodontic Irrigants

Endodontic irrigations can be characterized as shown in Figure (2.1) (Gomes *et al*., 2023; Middha *et al*., 2022).

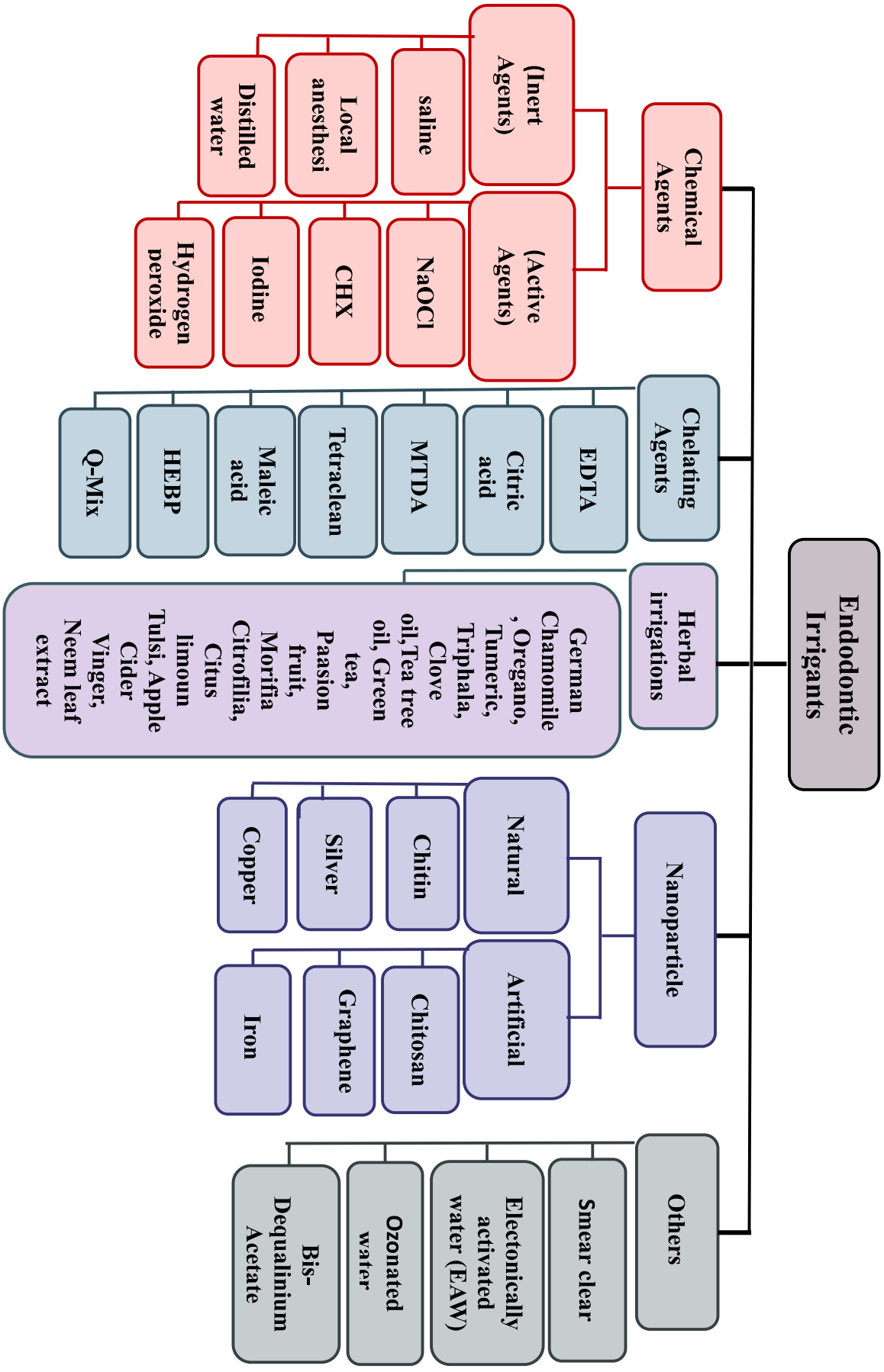

Figure (2.1): Classification of endodontic irrigants

2.6.1. Inert Agents

2.6.1.1. Distilled water

Water provides a good washing effect but is not a good disinfectant for endodontics. Bacteria with cell walls are frequently discovered in root canals after irrigation with distilled water. Chemical precipitates do not form when distilled water is combined with the majority of chemicals used in root canal therapy. As a result, it appears that the best irrigant for intermediate rinses to get rid of leftover chemical irrigants is distilled water. To prevent any interactions between the chemicals used in the root canal, it is crucial to eliminate all remnants of them (Gomes *et al*., 2023).

2.6.2 Active Agents

2.6.2.1 Sodium Hypochlorite (NaOCl)

It is the primary endodontic irrigant because of its extensive antibacterial action and capacity to break down organic materials. At body temperature, reactive chlorine in aqueous solution exists in two forms-hypochlorite (OCl–) and hypochlorous acid (HOCl). State of available chlorine depends on pH of solution, i.e. above pH of 7.6, it is mainly hypochlorite form and below this pH, it is hypochlorous acid. Presence of 5 percent of free chlorine in sodium hypochlorite is responsible for breakdown of proteins into amino groups. The pH of commonly used sodium hypochlorite is 12, at which the OCl form exits. Hypochlorite dissolves necrotic tissue because of its high alkaline nature (pH 12). To increase the efficacy of NaOCl solution, 1 percent sodium bicarbonate is added as buffering agent. Buffering makes the solution unstable, thus decreases its shelf life to even less than one week. Raising the temperature (45-65) and sodium hypochlorite concentration can greatly enhance the tissue dissolving capacity and debridement capabilities. Lowering the surface tension of NaOCl can improve the root canal system's capacity to penetrate the

uninstrumented area. Many advantages and disadvantages are there in table (2.1) (Abuhaimed and Abou Neel, 2017)

Table (2.1): Advantages and Disadvantages of NaOCl Irrigation (Kaur *et al.*, 2021).

Advantages	Disadvantages
1. Tissue dissolution and necrotic tissue 2. Remove an organic portion of dentin 3. Removes biofilim necrotic tissue 4. lubrication of canal 5. Antibacterial and bleaching action 6. Economical and easily available	1. Cytotoxicity causing excruciating pain, periapical bleeding, and swelling when extruded periapically. 2. High surface tension so its ability to wet dentin is less 3. Bleach clothes 4. Corrode instruments 5. Bad odor and taste 6. Not be used as a final rinse before obturation.

2.6.2.1.1 Effect of NaOCl in Dentine Composition

NaOCl solutions can also act in the dentin changing its chemical composition. In mineralized dentin, the collagen fibrils are encapsulated by apatite crystals, thus the dimensions of molecules that can penetrate in the dentin structure should be smaller. NaOCl molecules can penetrate in the apatite-encapsulated collagen matrix because of their low molecular weight (74.4 Da) and as a nonspecific oxidizing and proteolytic agent, can oxidize the organic matrix, denature the collagen, and adversely affects the mechanical properties of dentin. The effects of NaOCl solutions on the collagen of the dentin organic matrix may also affect the sealing ability and the adhesion of resin-based cements and root canal sealers that chemically bond to the dentinal collagen (Tartari *et al.*, 2016).

When sodium hypochlorite solution is the first irrigant used in the root canal, it has a weak ability to dissolve organic tissues, because the surface of the collagen is covered by hydroxyapatite from the smear layer. To dissolve this layer of hydroxyapatite, it is necessary to use a descaling agent. In this way, NaOCl can act directly on the underlying collagen, causing rapid destruction of collagen in the superficial dentin (Cai *et al.*, 2023; Dranceanu *et al.*, 2023).

2.6.2.2 Chlorohexidine

Chlorhexidine (CHX) is an alternative irrigant to NaOCl because of its broad-spectrum antimicrobial activity and considerably lower toxicity than NaOCl The most widely used concentration of CHX for root canal therapy is 2%. In contrast to NaOCl, high concentrations of CHX exert a bactericidal effect, whereas low concentrations provide only a bacteriostatic effect[21]. CHX can be used either as a gel or solution with the same effectiveness[15]. It exhibits the unique property of substantivity; the positive charges of the CHX molecule bind to the negative charges on dental surfaces resulting in prolonged adherence, which in turn leads to long-lasting antimicrobial activity. However, as an endodontic irrigant, the lack of tissue-dissolving capacity of CHX is a considerable drawback (Ruksakiet *et al*., 2020). Advantages and disadvantages of chlorohexidine are listed in table (2.2) (Sethi and Raji, 2021).

2.6.2.2.1 Tissue dissolving action of CHX irrigation

Chlorhexidine's capacity to dissolve tissue is negligible when compared to NaOCl. Periodontal therapy often uses solutions with a concentration of 0.1 to 0.2%, but root canal irrigating solutions with a concentration of 2% are typically seen in endodontic literature (Thakur *et al*., 2020).

Table (2.2): Advantages and Disadvantages of CHX Irrigation

Advantages	Disadvantages
1. 0.2% is used in controlling plaque 2. 2% is used as a root canal irrigant 3. It is more effective against gram-positive bacteria 4. Used with calcium hydroxide as intra-canal medicament (Hendy *et al*., 2024). 5. Act as intra-canal medicaments in necrotic tis-sue, retreatment cases and in vital pulp also because of its alkaline pH & also because of its antibacterial action,it can neutralize the remaining tissue debris in root canal.	1. It does not dissolve necrotic tissue 2. Does not show an effect on biofilms. 3. Less effective on gram-negative bacteria.

2.6.2.3 Ethylenediaminetetraacetic acid (EDTA)

EDTA is a calcium chelator that can remove minerals from the smear layer and debris on the root canal wall. The smear layer not only hinders the contact between the chemical irrigants and the root canal wall but also provides a living environment for the growth of bacteria. Clinically, EDTA is often applied in combination with NaOCl solution (≥2.5%) to remove the smear layer on the root canal wall. NaOCl can dissolve organic components and eliminate bacteria. EDTA forms a complex with calcium ions in hydroxyapatite to dissolve inorganic components, such as dentin debris, thereby cleaning the root canal wall and opening the dentine tubules, facilitating the chemical molecules in the irrigant to penetrate the dentin tubules to exert an antibacterial effect in the deep locations. EDTA unceasingly softens the root canal wall and should not be used as a final irrigation (Zou *et al*., 2024). Advantages and disadvantages are presented in table (2.3) (Kumar *et al*., 2021).

Table (2.3): Advantages and Disadvantages of EDTA

Advantages	Disadvantages
1. It dissolves dentin 2. It helps in enlarging the narrow root canal 3. Reduces time for debridement.	1. It does not dissolve inorganic dentin particles.

2.6.2.3.1 Mechanism of Action of EDTA Irrigation

The mechanism of action of EDTA based on their reaction with calcium ions to form soluble calcium chelates, which decalcify dentin. It has been reported that EDTA decalcified dentine to a depth 20-30 µm in 5 min. EDTA is normally used in a concentration of 17% and can remove the smear layers when in direct contact with the root canal wall for less than 1 minute (Doumani et al.,2017).

EDTA has high chelating capability which might be attributed to its increased ability to demineralize the smear layer, particularly the inorganic components. Different techniques may be used to enhance the action of EDTA, e.g., ultrasonic or sonic activation, negative pressure irrigation, laser activation, and manual dynamic agitation. At the same time, EDTA activation may also cause the surface disintegration of root canal dentin (Mikheikina *et al*., 2023). EDTA exerts only a weak antimicrobial effect but it seems to disrupt the biofilm matrix thereby promoting its detachment (Busanello et al., 2019), so it may also supplement the anti-biofilm effect of NaOCl. However, EDTA activation may also cause surface disintegration of root canal dentin. Repeated use of EDTA triggered root canal dentin erosion, decalcification of the peritubular dentin and dissolv-ing of the exposed organic matrix (Boutsioukis and Arias-Moliz, 2022; Gawdat and Bedier, 2022).

Collagen opens and micro-hardness diminishes as a result of chelation and demineralization agents dissolving the calcium hydroxyapatite matrix from dentin in addition to the inorganic structure in the smear layer (Bayram *et al*., 2017).

2.6.3 Nanoparticles

Recently, a growth in nanotechnology have been seen because of its potential to improve system performance. Numerous researches have investigated how nanotechnology is being applied in engineering domains (Ahmadi *et al*., 2018). Nanotechnology is not only useful in engineering systems but also in medical sciences. The use of nanotechnology can make better medical care possible. Tools are more suitable for use in medical devices because of nanotechnology, which offers the potential to reduce device size and increase material strength (Jiang *et al*., 2018; Ahmadi *et al*., 2019).

The word "Nano" originates from the Greek word "dwarf.". There are numerous sizes and forms of nanoparticles. Nanoparticles vary in size from 1 to 100 nm. Among their many traits are their incredibly small diameters

and increased chemical activity (Ibrahim *et al*., 2017). Because of their biocidal, anti-adhesive, and transport qualities, nanoparticles are widely used in infection control measures, especially in the complex environment of the mouth cavity. When compared to their conventional counterparts, the most beneficial elements in antibacterial behavior are the larger surface areas of the nanoparticles and the higher concentrations at the target region (Bhardwaj *et al*., 2014; Zhang *et al*., 2010). Anti-dhesive meaning inhibit the attachment of cells, bacteria, or other materials to a surface. This is achieved through various mechanisms, including surface modification, the release of antimicrobial agents, or by altering the surface's physical properties like hydrophobicity (Saravia et al., 2020).

Nanoparticle-sized materials are produced via nanotechnology and they possess unique physicochemical properties such as high surface area and amplified chemical reactivity. This leads to a higher concentration of atoms near the surface, in comparison to their microscale or macroscale-sized counterparts (Priyadarsini *et al*., 2018; Ragab *et al*., 2022).

Lately, natural biomaterials are introduced in endodontics to restore structural strength that is destroyed by synthetic chemical solutions like CNP that limit bacterial penetration, prevent dentinal micro-fractures, and improve the mechanical properties of the root dentin. Because of its greater capability of chelating for various metal ions in root dentine and low price, it was a preferable irrigant for the study (Nirmal *et al*., 2022; Tekin and Demirkaya, 2020)

Nanoparticles possess unique physicochemical properties due to their petite sizes, elevated surface area-to-mass ratios, and enhanced chemical response (Marica *et al*., 2022; Raura *et al*., 2020).

The use of irrigants based on nanoparticles has become more common due to the limitations of traditional irrigation techniques. The use of nanoparticles in the diagnosis and treatment of oral illnesses to enhance

general oral health is known as "Nano dentistry". Since there are various nanomaterials that have antimicrobial properties, nanotechnology can be applied to endodontics to improve the quality of disinfection and treatment procedure as figure (2.2) shows (Akbarianrad *et al*., 2018; Chandak *et al*., 2021).

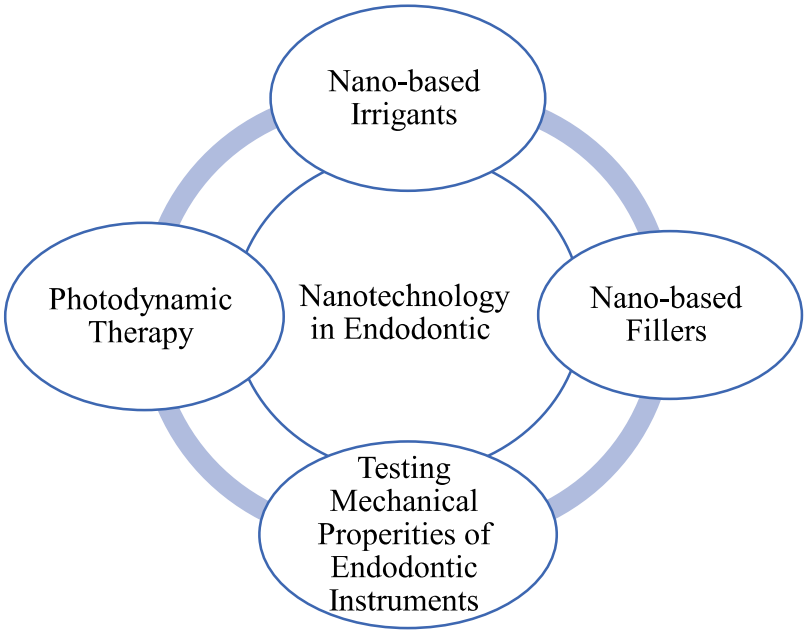

Figure (2.2): Application of Nanotechnology in Endodontic (Akbarianrad *et al*., 2018)

2.6.3.1 Synthesis of Nanoparticles in Dentistry (Abid *et al*., 2022)

1) **Top-down approach**: In this approach, the bulk of the material is made to shrink to a nanoscale structure with specialized treatments like grinding, ablation, etching, and sputtering.
2) **Bottom-up approach**: In this latter approach the material is made to undergo chemical reactions. Methods utilize organo-metallic chemical route, revere-micelle route, sol-gel synthesis, colloidal precipitation, hydrothermal synthesis, template-assisted sol gel, and electrodeposition.

2.6.3.2 Classification of Nanoparticles

Nanoparticles in dentistry can be categorized according to different characterizes as in figure (2.3) (Bhushan and Maini, 2019; Shrestha and Kishen, 2016; Yahya & Jamel, 2023).

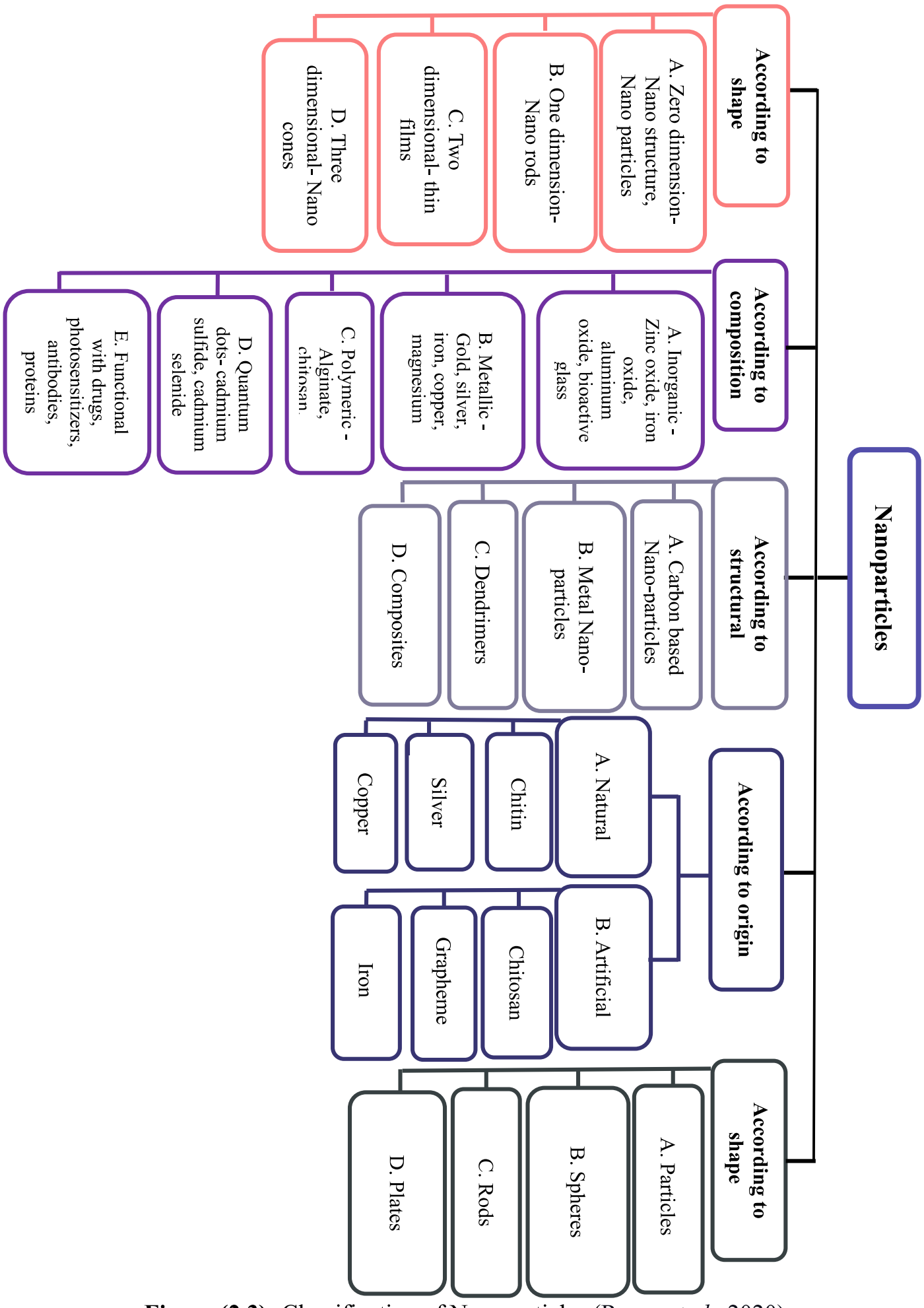

Figure (2.3): Classification of Nanoparticles (Raura *et al*., 2020).

2.6.3.3 Chemical Structure of Chitin and Chitosan

Chitin (CH) is considered the second most abundant polysaccharide after cellulose This natural amino-polysaccharide copolymer is the building block of the exoskeleton of marine crustaceans giving them durability and stability against the natural forces (Elieh-Ali-Komi and Hamblin, 2016; Madni *et al*., 2021; Ibrahim *et al*., 2023).

Chitosan is a semi-crystalline polysaccharide consisting of linearly arranged N-acetyl-glucosamine and d-glucosamine residues. Its structure contains an amino group (-NH2), which makes it cationic (Khan and Alamry, 2021). This positive charge facilitates the formation of the extracellular matrix by drawing negatively charged molecules like proteoglycans. Additionally, a hydroxyl group (-OH) is present in the structure, which attracts positively charged molecules to improve bonding. In addition to electrostatic attraction, these functional groups help modify chitosan, improving its mechanical and physical properties and conferring novel functional characteristics and compelling clinical relevance, figure (2.5) (Herdiana *et al*., 2022).

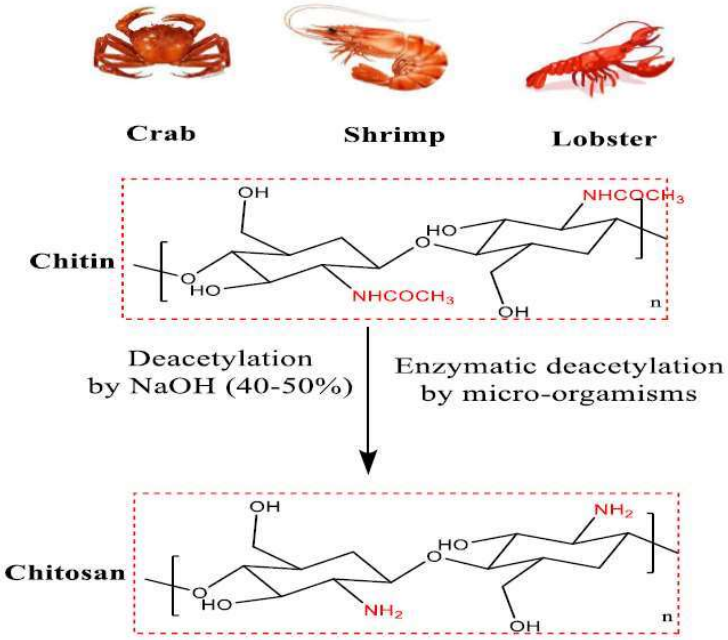

Figure (2.4): Chemical structure of chitin and chitosan

2.6.3.4 Chitosan Nanoparticles

Chitosan has attracted attention in dental research because chitosan is a naturally occurring polysaccharide that is non-toxic, biocompatible, biodegradable, and bioadhesive. Chitosan, which is made via chitin deacetylation and is found in shrimp and crab shells, is abundant in nature and has a cheap cost of manufacture. Because of this, it is an environmentally friendly material that may be used for a range of purposes (Al-Ogaidi, 2021).

Chitosan is a positively charged polyelectrolyte with a pH range of 2–6, which results in its greater solubility when compared to chitin. This characteristic makes chitosan solutions extremely viscous thus difficult to manipulate. This is one of the limitations but chitosan solution affects pressure distribution because chitosan has a D-glucose structure and has the the ability to strengthen dentine on the root canal wall (Naveed *et al*., 2019; Liu *et al.,* 2020).

Chitosan may be a useful substitute for many traditional root canal irrigations, either alone or in mixing with other irrigation techniques. Additionally, chitosan's high chemical activity and functionality allows it to be combined with other substances to increase its antibacterial properties. For example, chitosan can be added to calcium hydroxide, zinc oxide, eugenol, and some types of photosensitizer to increase these materials' antibacterial effectiveness against endodontic pathogens (Simanjuntak *et al*., 2019; Dragland *et al*., 2019; Wong *et al*., 2021).

Chitosan nanoparticles are soluble in weak acid solutions and could be also soluble in water. They are employed in dentistry to eradicate bacteria and remove the smear layer due to their deeper absorption and penetration into dentinal tubules and intertubular dentine penetrates the intricate root canal and dentinal tubules (Bertani *et al*., 2021).

Numerous studies have demonstrated that chitosan functions as a chelator and may enhance dentine wetting. At the same time, chitosan nanoparticles showed that they could protect dentine tissue's collagen by stopping bacteria from breaking down the collage. Applications of chitosan

in dentistry are illustrated in figure (2.4) (Hashmi *et al*., 2019; Agrawal *et al*., 2023; Arora *et al*., 2023; Paradowska-Stolarz *et al*., 2023)

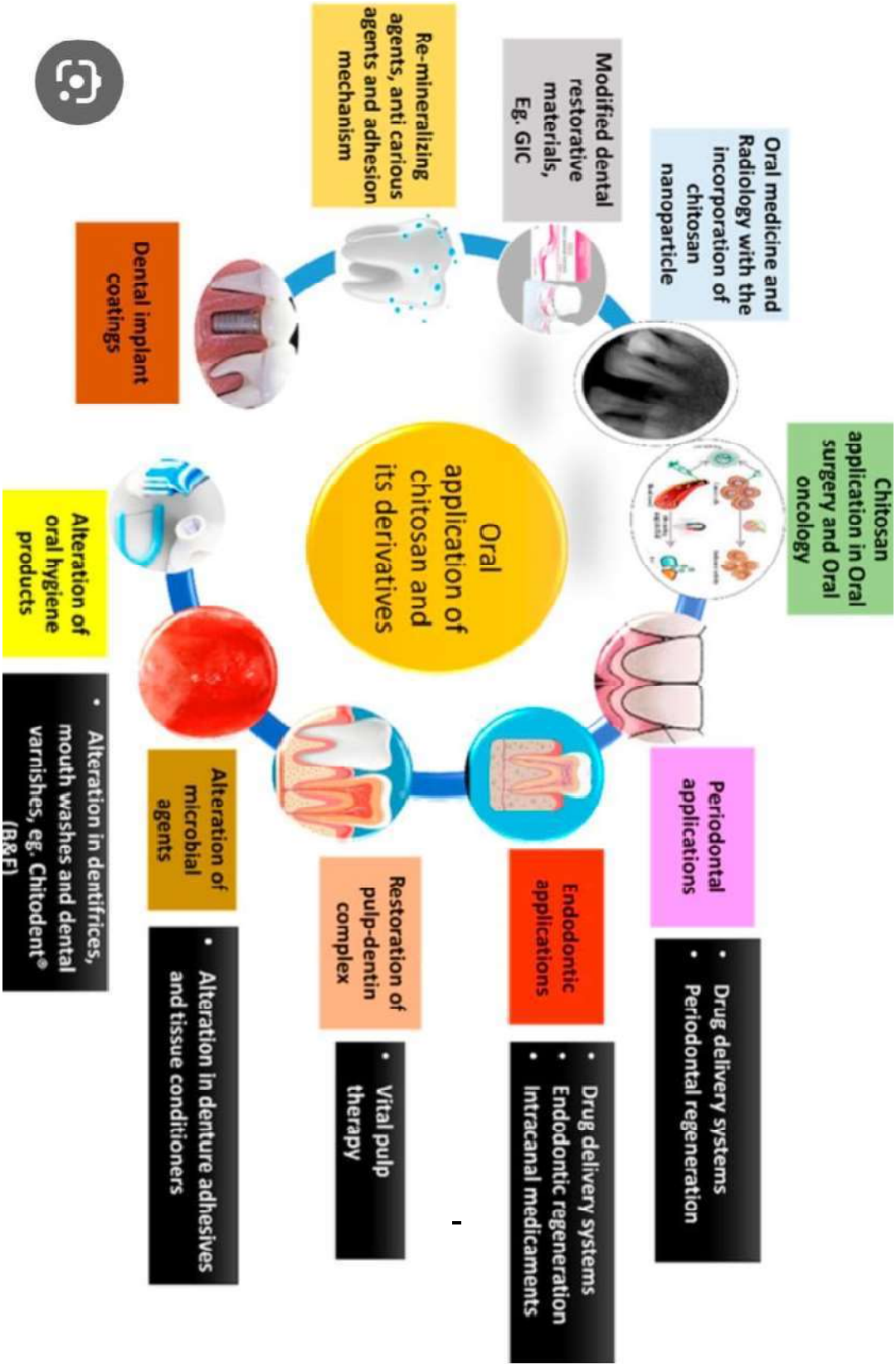

Figure (2.5): Application of chitosan in dentistry (Mascarenhas *et al*., 2024).

2.6.3.5 Commonly Used Methods in Preparing Chitosan

In general, the methodologies published can be categorized into two types: chitosan extracted from the crustacean by-products via chemical methods, and via biological methods (enzymatic method and fermentation method), figure (2.6) (Kou *et al*., 2021). Different techniques have been developed to produce CNPs just like Ionotropic Gelation, ionotropic gelation with the radical polymerization method, emulsion droplet coalescence, emulsion solvent diffusion, reverse micellisation, desolation, nanoprecipitation method and spray drying method (Bashir *et al*., 2022).

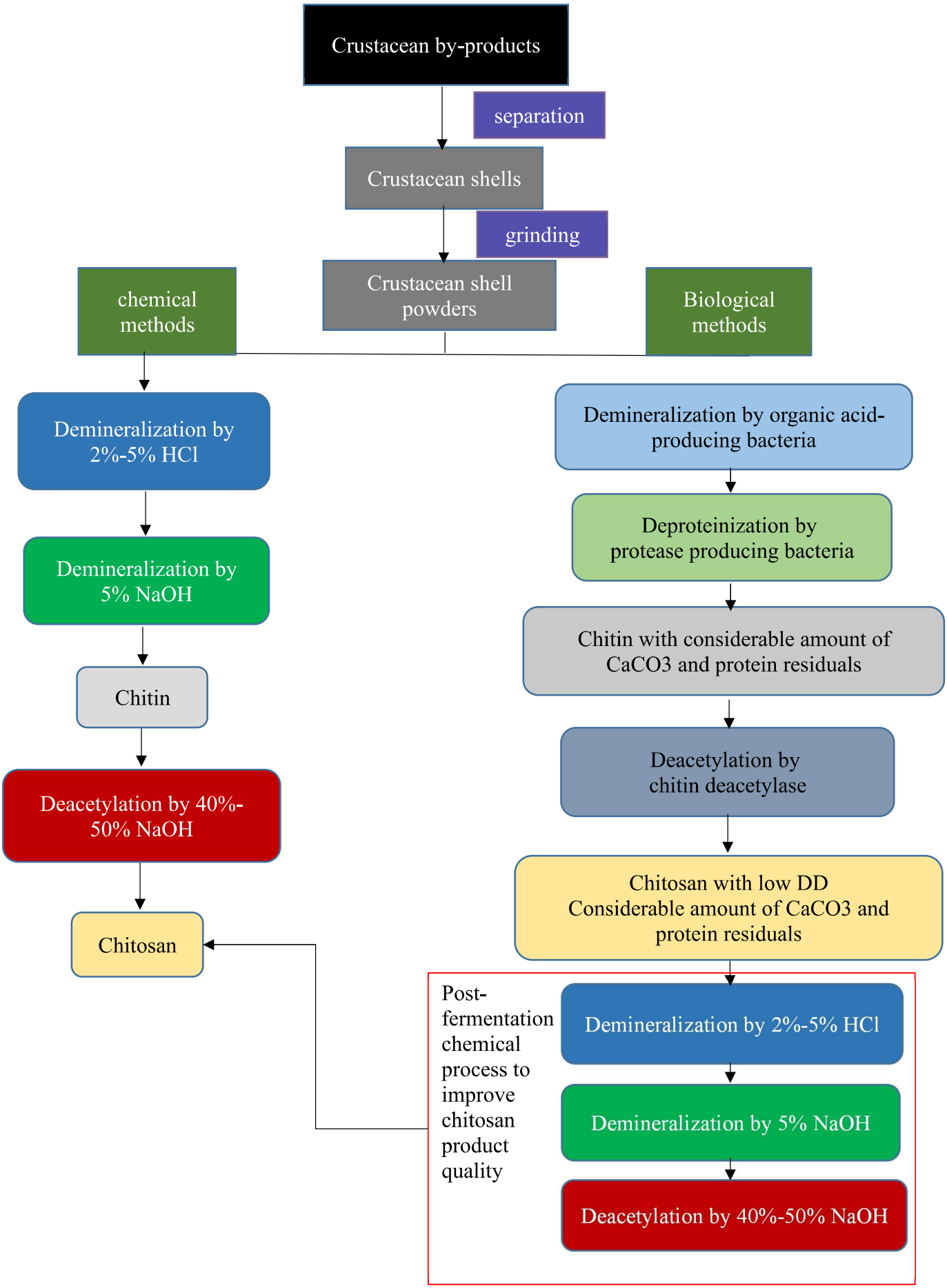

Figure (2.6): Chemical methods of preparing chitosan from seafood byproducts as compared to biological methods of fermentation (Kou *et al*., 2021).

2.6.3.6 Chelation Mechanism of Chitosan

There are several theories related to the chelation mechanism of chitosan to dentinal structure.

1. The first, known as (bridge model theory), proposed that two or more amino groups in a chitosan chain bind to the same metal ion (Ferreira-Reguera *et al*., 2025).
2. The second, known as (metal ion) is bound by a single amino group (Ferreira-Reguera *et al*., 2025).

Due to chitin's unbound pairs of electrons and nitrogen atoms, which make up the chitosan polymer, ionic contact between the metal and the chelating agent occurs. The processes of ion exchange, adsorption, and chelation lead to the formation of complexes between chitosan and metal ions. The kind of contact that occurs is determined by the ions' kind and the chemical structure of chitosan (Abu Al-timan *et al*., 2019)

Chitosan exhibits fewer alterations to the dental hard tissue's structure than EDTA. These findings confirm that, in comparison to EDTA, chitosan is a weak chelating agent that demineralizes less dentin surface. As a result, smear layer removal using chitosan nanoparticle solution has been successful, although dentin demineralization has not been induced. Furthermore, remineralization of demineralized dentin is believed to be induced by chitosan, which comes into touch with the dentin surface. It has been postulated that the covalent bond between chitosan and dentin collagen produces mineralization of demineralized dentin. This phenomenon, known as the calcium-phosphate layer, is caused by phosphate groups that have that can draw calcium ions and create a surface suitable for crystal nucleation. Many advantages and disadvantges of chitosan nanoparticles are there as in table (2.4) (Gusiyska *et al.,* 2016; Kishen and Shrestha, 2018)

Table (2.4): List of advantages and disadvantges of CNPs (Grag *et al.*, 2019).

Advantages	Disadvantges
1. Less toxicity 2. Biocompatibility 3. Mucoadhesive character 4. Stability 5. Drug targeting is site-specific 6. Therapeutic index of the drug is increased 7. Frequent, expensive and unpleasant dosing is prevented 8. Color compatible to tooth structure. 9. Cost effective. 10. Availability. 11. Ease of chemical modification	1. Mechanical resistance is less 2. Difficulty in controlling pore size 3. May contract 4. stability Electrospinning is difficult for pure chitosan 5. Preparation by crosslinking can affect intrinsic properties of chitosan 6. Low solubility in neutral and alkaline pH 7. Method of preparation must be changed according to the drug to be delivered

Table (2.5): Characteristics of Endodontic Irrigants

NaOCl(Nogo-Zivanovic *et al.*, 2018)	• effective antimicrobial agent • organic tissue solvent • lubricates • toxic • corrosive effect • not substantive • removes only the organic part of the SL
CHX(Nogo-Zivanovic *et al.*, 2018)	• wide range of antimicrobial effects against G + and Gbacteria and fungi • substantively in dentin for up to 12 weeks • dentin components, inflammatory exudate may inhibit the antibacterial activity • no ability to dissolve organic or inorganic tissue • biocompatibility
EDTA(Nogo-Zivanovic *et al.*, 2018)	• effectively removes the SL after the initial NaOCl irrigation • demineralization of dentin
Chitosan (Raura *et al.*, 2020)	1. Inactive and non-soluble in water alkali and organic solvents, pH more than 6 3. Soluble in various other mediums 4. Highly viscous, with a polyelectrolyte property. 5. It is a linear polyamine. 6. The presence of highly reactive hydroxyl and amino groups results inhalation of various transitional metal ions 7. Excellent antibacterial, antifungal and antiviral properties. 8. Causes disruption of the bacterial cell membrane due to its electrostatic interaction.

2.7 Root canal sealer

Maintaining a sterile environment in the pulpal space is the last step in endodontic treatment, which involves filling the root canal with a material that is inert, biocompatible, and has antimicrobial and physicochemical properties, and securing it with a final restoration. All of these steps are necessary to ensure the long-term success of endodontic treatment. The process begins with an accurate diagnosis and treatment planning (Jo *et al*., 2020; Mendes *et al*., 2018).

Diverse endodontic methods have been proposed for the obturation of the radicular space. The majority utilize a core substance and a sealant. Every procedure needs a sealer, regardless of the type of core used; it creates a fluid-tight seal by assisting in the bonding process between the root canal wall and the core. In addition, sealers function as lubricants throughout the obturation procedure and occupy voids and abnormalities in the root canal, lateral and auxiliary canals, and interstitial spaces between gutta-percha sites. Finally, they entomb the remaining surviving microbes and starve them of nutrition, all of which can be significant sources of future infections (Reszka *et al*., 2016; Khalil *et al.,* 2019; Thejeswar and Ranjan, 2020).

A root canal sealer needs to have the right physicochemical and biological characteristics. The periradicular tissues shouldn't be irritated by it. Stimulating biologic sealing and restoration through mineralized tissue deposition in the apical foramen would be ideal (Abu Zeid and Alnoury, 2023; Jafari and Jafari, 2017; López-García *et al*., 2020).

2.8 Ideal Properties of Root Canal Sealer

Grossman defined the requirements for the perfect sealer in (1988). But no sealer has met all the requirements as of yet (Rathi *et al*., 2020)

1. It should offer improved adhesion and be sticky when combined.
2. It ought to offer a fluid tight hermetic closure.

3. It should be radiopaque.
4. Its particle size should be modest to facilitate simpler mixing.
5. It shouldn't get smaller when set.
6. The tooth cannot be discolored by it.
7. Antimicrobial properties.
8. It shouldn't dissolve readily in bodily fluids.
9. It shouldn't shrink after setting.
10. Non-irritating and biocompatible with periodontal or periapical tissue.
11. It should dissolve readily in common solvents so that retreatment instances can be handled with ease.

Few other requirements that can be added to Grossman's (11) basic requirements are (Mishra *et al*., 2018):

12. It should not provoke an immune response in periradicular tissue.
13. It should be neither mutagenic nor carcinogenic.
14. It should be capable of bonding to dentin or gutta-percha / core obturation material.

2.9 Root Canal Sealer Classification (Liu *et al*., 2019; Zhekov and Stefanova 2020)

1. Zinc Oxide Eugenol Based Sealers
2. Calcium Hydroxide Based Sealers
3. Glass Ionomer Based Sealers.
4. Epoxy Resin Based Sealers
5. Silicone Based Sealers
6. Bioceramic Based Sealers

2.9.1 Zinc Oxide Eugenol based Sealer (ZOE)

The formula of these sealers created by Grossman in (1936) became Proco-Sol sealer (StarDental, Lancaster, PA, USA), and the formulation created by Rickert and Dixon in (1931) became Kerr sealer. Because of their

long-term performance, ZOE sealers have been the industry standard in endodontics since their inception. Eugenol liquid, an essential oil made from cloves, and zinc oxide powder are the ingredients of ZOE sealers. Even though eugenol in ZOE sealers has been shown in ex vivo laboratory experiments to have cytotoxic characteristics, these sealers are clinically helpful in both animal and human investigations. Eugenol and zinc oxide combine to create an amorphous gel when applied to moist root dentin. A stiff matrix is created in the gel by the residual zinc oxide powder. The powder component of some of these powder-liquid sealers contains silver, which has resulted in tooth discoloration. To solve this problem, formula that does not stain is created that includes no silver (Komabayashi *et al*., 2020).

Conventional (ZOE) sealers are still in use because of their slow set, low cost, antibacterial qualities, and simplicity of application. Its shortcomings include weakening the root structure, not adhering to the dentin, microleaking, high solubility, periapical tissues being cytotoxic and irritable even after setting, and being weak and porous after setting, which leaves them vulnerable to breakdown if tissue fluids leak. They could all affect how long root canal therapy lasts (Johnson *et al*. 2021; Rathee *et al*., 2020).

2.9.2 Epoxy Resin Based Sealers

There are two types of resin sealers: methacrylate and epoxy resin-based sealers. Resin sealants possess a longstanding history of application due to their adhesive properties. The highly sought-after capacity of this specific brand of bondable root canal sealer to form monoblocks inside the root canal space has led to extensive marketing. Their enhanced flow, capacity to infiltrate dentinal tubules, robust adherence to dentin, a core obturating substance with high sealing capability, and excellent apical sealing with minimal microleakage all work to fortify the weak root. They also have poor solubility, robust radiopacity, and good dimensional stability. Among root canal sealers, these characteristics are thought to be crucial. In the event of

retreatment within a deep and narrow root canal, these sealers have issues with bonding and/or removal (Arikatla *et al.,* 2018; Garg *et al.,* 2019; Komabayashi *et al*., 2020; Rathee *et al*., 2020; Rekha *et al*., 2023).

Among currently available root canal sealers, resin-based sealers represented by AH Plus are the most widely used. AH Plus is an epoxy resin-based endodontic sealer, available in a paste-paste form. Several studies have considered AH Plus as the gold standard, given its excellent physical properties and sealing ability, due to its resorption resistance and dimensional stability. However, it has limitations, such as possible mutagenicity, cytotoxicity, and an inflammatory response. In addition, its hydrophobicity prevents the complete filling of the hydrophilic canal. Specifically, defects in AH Plus adhesion to the canal walls can occur due to retained dental moisture (Camargo *et al*., 2017; Lim *et al*., 2020).

However, these sealers' major drawback is their missing of bioactive qualities. Various degrees of cytotoxicity for these sealers were shown in laboratory and animal tests. Others observed its poor sealing capacity because of its increased water solubility and polymerization shrinkage, which can cause gaps to emerge in the resin-dentin link (Colombo *et al*., 2018; Fonseca *et al*., 2019; Dioguardi *et al*., 2021; Souza *et al*., 2023).

2.9.3 Bioceramic sealers

Ceramic materials created especially for dental and medical applications are known as bioceramics. For the past thirty years, bioceramic-based sealers have only been accessible for use in endodontics; their rise to prominence has coincided with the growing usage of bioceramic technology in the medical and dental disciplines. These consist of calcium phosphates, zirconia, hydroxyapatite, glass ceramics, bioactive glass, and alumina (Al-Haddad and Che Ab Aziz, 2016; Al-Sabawi and Al-Jubori, 2024; Baghdadi *et al*., 2021; Jo *et al*., 2020; Prashanth, 2017).

Based on how bioceramic interacts with the surrounding living tissues, it can be divided into the following categories (Dong and Xu, 2023; Patel *et al*., 2022):

1. Bioinert materials, such as Alumina and zirconia, do not interact with biological processes.
2. Biodegradable materials, such as tricalcium phosphate and bioactive glasses, may be soluble or resorbable and eventually absorbed or replaced into a tissue.
3. Bioactive materials, such as bioactive glasses, bioactive glass ceramics, hydroxyapatite (HA), and calcium silicates, can interact interfacially with the surrounding living tissues.

The precise process by which the bioceramic-based sealer adheres to the root dentin is unknown; however, the following mechanisms for calcium silicate-based sealers have been proposed (Al-Haddad and Che Ab Aziz, 2016; Chellapandian *et al*., 2022; Zhekov and Stefanova, 2021):

1. Tubular diffusion is the process by which the sealer particles diffuse into the dentinal tubules to create mechanical interlocking linkages.
2. Denaturing the collagen fibers with a strong alkaline sealer causes the sealer's mineral content to pass into the intertubular dentin and create a mineral infiltration zone.
3. Calcium silicates react with the moisture in the dentin to form calcium silicate hydrogel and calcium hydroxide, which partially reacts with phosphate to form hydroxyapatite along the mineral infiltration zone.

Bioceramic sealers are Biocompatible, both its mineralization process and its bactericidal activities are enhanced by high alkalinity (pH 12.8) during setting.

Clinical disadvantages of bioceramic-based sealers include handling challenges, increased cytotoxicity when mixed fresh, prolonged setting times, high solubility, comparatively high costs, the requirement for

sufficient moisture to cause hardening, and difficult to remove in retreatment cases (Walsh *et al.,* 2018; Donnermeyer *et al*., 2019).

Furthermore, because endodontic bioceramic materials are not impacted by moisture or blood contamination, they are method-independent. Bioceramic materials also have the advantage of promoting the formation of HA (bioactivity), which in turn helps the dentin and filling material to bond together during setting, leading to improve the sealing ability and supporting the regrowth and repair of the damaged periapical tissue (Estivalet *et al*., 2022).

Various brands of calcium based root canal sealers are available on the market. These can be classified according to their main components (Alaenazi *et al.,* 2018; Sfeir *et al.,* 2021).

A. Mineral trioxide aggregate-based sealers.

B. Calcium phosphate-based sealers.

C. Calcium silicate-based sealers.

D. Bioactive glass-based sealers.

A. Mineral Trioxide Aggregate-Based Sealers (MTA)

The first type of bioceramic material is an MTA. Torabinejad created it in the early (1990s). Tricalcium silicate, dicalcium silicate, tricalcium aluminate, tetracalcium aluminoferrite, bismuth oxide or zirconium oxide, and silicate oxide are the primary ingredients of MTA. The physical and chemical characteristics of MTA are also attributed to a few other mineral oxides (Lim *et al.,* 2020).

MTA, when used as a sealer, generates Ca(OH)2, which forms an interstitial layer resembling HAP structures in simulated bodily fluids. This, in turn, causes the major apical foramen to close due to cementum deposition. MTA is activated with the introduction of water and produces a highly alkaline mixture with a pH of 12 and beneficial hydrates of calcium silicate. These hydrates make MTA an excellent endodontic material as they

form on the surface of existing calcium silicate particles and continue to grow inwards, creating a barrier effectively (Jitaru *et al*., 2016; Tanomaru-Filho *et al.,* 2016; Raghavendra *et al*., 2017).

High alkaline pH (10–12.5), high biocompatibility, hydrophilicity, osteoinductive, and osteoconductive characteristics promoting mineralization, bioactivity, antimicrobial efficacy, insoluble over time, resistance to moisture and blood contamination, and good adhesive properties to the root dentin that enhances interfacial adaptation and hermetic seal are all characteristics of most MTA brands used as root canal sealers in endodontics (Rathod, 2023).

Among its many drawbacks are the possibility of discoloration, a long setting time, a short working time of less than four minutes, poor handling qualities, low compressive strength, and challenges in removing the MTA from root canals when retreatment is needed (Al-Sabawi and Al-Jubori, 2024).

MTA Fillapex has been developed to combine the physicochemical properties of a resin-based root canal sealer with the biological properties of MTA. MTA fillapex has a high flow rate (27 mm) and a thin film, it easily penetrates the other accessory channels. Regardless of the sealing technique, MTA fillapex confidently provides a high level of sealability that is not affected by heat, unlike other seals. Furthermore, it has a high PH value for a long-lasting antibacterial effect and can maintain a relatively constant calcium release for up to 14 days. MTA Fillapex has the advantage of being easy to handle and resistant to resorption but its drawback is that it lacks solubility and staining (Khandelwal and Ballal, 2016; Sharma *et al*., 2023).

2.10 Scanning Electron Microscope

Scanning electron microscope (SEM) and transmission electron microscope are the important analytical approaches that can be used to describe nanoparticles and porous materials.

The SEM is a prevalent technique for high-resolution surface imaging, which is capable of characterizing materials at the nanoscale and microscale. SEM analysis has proven to be more successful in the examination of inorganic nanoparticles within complicated biological systems. To assess the dimensions, morphology, and size distribution of nanomaterials, SEM analysis only requires basic sample preparation, such as drying and conducting the samples (Mourdikoudis *et al.,* 2018).

In Scanning Electron Microscopy (SEM) the beam is focused on the sample and scanned over a defined area of the sample. With the use of electrostatic or electromagnetic lenses and a faster electron beam, the SEM may attain a deeper field of vision and a higher magnification of 100,000X. The high-energy electrons produced by SEM, which are concentrated at the surface of solid materials after their interaction with the sample, produce a range of signals that reflect the atomic composition and topographical properties of the surface. Once the incident electron beam has scanned the sample's surface, an electron detector measures the released electrons for different points within the scanned area. After displaying the brightness dictated by the intensity of the emitted electrons, the morphology of the sample surface is captured as a digital image (Al Thaher *et al*., 2022).

2.11 Push Out Bond Strength Test

One of the key characteristics of endodontic sealants is their strong dentin bond. The simple, geometrically defined bond's cross-sectional area divided by the initial mechanical load to fracture is the definition of bond strength (El Mourad, 2018).

The bond strength test is the most efficient way to gauge how well a root canal filling adheres to surfaces, though there are other techniques as well. One of these techniques, the push-out test, has been widely used to evaluate the filling materials' resistance to dislodgement, the strength of the sealer-dentin bond, and the retention of posts luted in root canals. It is simple

to carry out, decipher, duplicate, and record (Tsintsadze *et al*., 2017; Nafiz *et al.,* 2018; Dem *et al.,* 2019).

The possibility of placing the sealer in direct contact with the dentin walls inside the canal rather than on the flat surface of coronal dentin, which has a different tubular arrangement pattern, is a one advantage of this method. As a result, the sealer material adapts to the shape of the canal and penetrates inside the dentinal tubules, providing mechanical retention comparable to that of clinical conditions. Additionally, because this test causes breakage parallel to the dentin-sealer interfacial surface, more reliable and clinically effective results are obtained, better illustrating the strength of the sealer's bond strength (Brichko *et al.,* 2018; Jain *et al.,* 2019).

When setting up the test, several aspects must be considered as they can affect the validity of results. These variables include the qualities of the root-filling material, the widths of the root canals, the orientation of the sample, and the size of the plunger utilized. Consequently, the force is applied to dentin rather than filling materials when the plunger diameter exceeds the canal space, yielding significantly better outcomes. Conversely, a smaller plunger diameter about the canal diameter may result in reduced bond strength and filling material punctures. This reading will serve as a representation of the filling's strength (Brichko *et al.,* 2018; Nafiz *et al.,* 2018).

CHAPTER THREE

MATERIALS AND METHODS

CHAPTER THREE

MATERIALS AND METHODS

3.1 Materials, Equipment, and Instruments Utilized in This Study

3.1.1 Materials

The materials utilized in this study are listed in table (3.1) and compositions of materials are list in table (3.2).

Table (3.1): Materials utilized in this study

No.	Materials	Manufacture
1.	Acetic acid glacial	Scharlau, Spain
2.	Chlorohexidine 2%	AQUA Medical, Sultanghazi, Istanbul, Turkey
3.	Cold cure acrylic resin	China
4.	Distilled water	Ninava Drug Industry, Mosul, Iraq
5.	Ethyl alcohol (30,50,70,90,100%)	Kair Al-joud, Iraq
6.	Ethylenediaminetetraacetate 17%	Imicryl, TurKey
7.	Flowable composite	Coltene, North American
8.	Nano chitosan powder (78 nm)	Shaanxi Sangherb Bio-Tech Inc CO., Ltd, China
9.	Pink wax	Polywax, Turkey
10.	Polysiloxane elastomer impression material	Zetaplus, Zhermack, Badia Polesine, Italy
11.	Polystone	Bilklim Tibbi Urunler Ltd. Sti, Izmir, Turkey
12.	Single bond universal adhesive	3M ESPE, Germeny
13.	Sodium hypochloride 2.5%, 3%	AQUA Medical, Sultanghazi, Istanbul, Turkey.

Table (3.2): Compositions of sealers

No.	Materials	manufacture	Compositions	Expire Date
1.	AH Plus sealer	Dentsply, Germany	Paste A: Bisphenol-A epoxy resin, Bisphenol-F epoxy resin, calcium tungstate, zirconium oxide, silica, iron oxide pigments. Paste B: dibenzyldiamine, aminoadamantane, tricyclodecane-diamine calcium tungstate, zirconium oxide, silica and silicone oil.	7/2024
2.	MTA Fillapex sealer	Angelus, Brazil	Base paste: salicylate Resin, natural resin, calcium tungstate, nanoparticulated silica, pigments. Catalyst Paste: diluting resin, Mineral Trioxide Aggregate, silica and pigments.	10/2024
3.	EndoFill sealer	PD Swiss, Switzerland	Zinc oxide, hydrogenated resin , bismuth subcarbonate, barium sulfate, sodium borate, eugenol and sweet almond oil.	12/2025

3.1.2 Equipment and Instruments

The Equipment and Instruments utilized in this study are listed in table (3.3) below.

Table (3.3): Equipment utilized in this study

No.	Equipments	Manufacture
1.	30 gauge Disposable Irrigation Needle	SinaliDent, China
2.	Barbed Broach	VDW GmbH, Germany
3.	Bench Vice	China
4.	Chisel	Martin, Germany
5.	Contra-Angle Slow Speed Handpiece	NSK NAC, Japan
6.	Dental bond brush	China
7.	Dental Micromotor	Strong 204, South korea
8.	Dental scaler	Guilin Woodpecker medical instrument CO.,LTD.; China.
9.	Dental Surveyor	Paraline, Germany
10.	Diamond disc 0.2mm	NEXUS MEDODENT, Korea
11.	Digital Stereomicroscope	Optika, Italy
12.	Digital Universal Testing Machine	GESTER, China
13.	Disposable syringe	EASYMED, China
14.	Ecotestr Ph2 Meter	Oakton, India
15.	Eighteeth E-connect Cordless Endo-motor	Changzhou Sifary Medical Technology Co., Ltd, China
16.	Electronic digital caliper	Jeyssui, China
17.	Electronic sensitive balance	METTLER TOLEDO, Swtizerland
18.	Endodontic ruler	Diadent, Korea
19.	Endo-motor	Eighteenth, China
20.	Glass beaker	ISOLAB, Germany
21.	Gloves	OnePlus, china
22.	Gutta-percha	Diadent, South Korea

23.	Incubator	EN400; Belgium.
24.	K-files (10 mm)	MANI, INC., Japan
25.	Lentulo spiral size 30	Tg, Germany
26.	Magnetic stirring machine	Daihan Labtech Co., Ltd, China
27.	Masks	China
28.	Microscope micrometer	Olympus, Japan
29.	Paper point	Diadent, South Korea
30.	Protaper Files Superfiles III NiTi heat activation system	Denco Medical CO., Ltd, China
31.	PVC pipe	China
32.	Scanning electron microscope	ZEISS, version 7.01, Germany
33.	Silicon rubber mold	China
34.	Slow speed straight handpiece	Being foshan medical equipement Co. Ltd., Guandong, China
35.	Sputtering device	Qurum, Q150R Plus, England
36.	Towels	China

3.2. Methods

3.2.1 Ethical Statement

Prior to the performance of the current study, ethical approval was obtained from the Research Ethics Committee at the College of Dentistry, University of Mosul, Mosul, Iraq. Ethical consent was gained for the whole set of tests conducted in this study with REC reference no. UoM.Dent. 23/73 on November 28, 2023.

3.2.2. Preparation of Chitosan Nanoparticle Irrigation Solution

The preparation of 0.2% of chitosan nanoparticle solution was aggregated by dissolving 2 gm of nano chitosan powder (78 nm) and was weighed up in a high-precision portable balance in V:100 ml of 1% acetic acid in a glass beaker. A magnetic stirring machine was employed for stirring the mixture within 2 hours at room temperature (±23°C) till a crystalline homogenous solution with (PH 3.8) (Mohamed Nour ElDeen *et al*., 2023).

To prepare 0.5% chitosan nanoparticle solution. About 1 ml of glacial acetic acid was added to 100 ml of distilled water to obtain 1 % of acetic acid in a glass beaker. Then 5 gm of Nano Chitosan powder (78 nm) was weighed up in high precision balance and added to a solution above 1% acetic acid. The mixture was agitated using a magnetic stirrer to produce a viscous solution for 2 hours at room temperature (±23°C) (PH 4.1) (Figure 3.1) (Mohamed *et al*., 2020). PH was determined by Ecotestr PH2 meter (Figure 3.2). The solution was saved in the refrigerator and used within two weeks after preparation (Bastawy and Ahmed, 2022).

Figure (3.1): Preparation of CNP irrigation (A) Acetic acid glacial, (B) Nano chitosan powder, (C) High precision balance, (D) Magnetic stirrer machine.

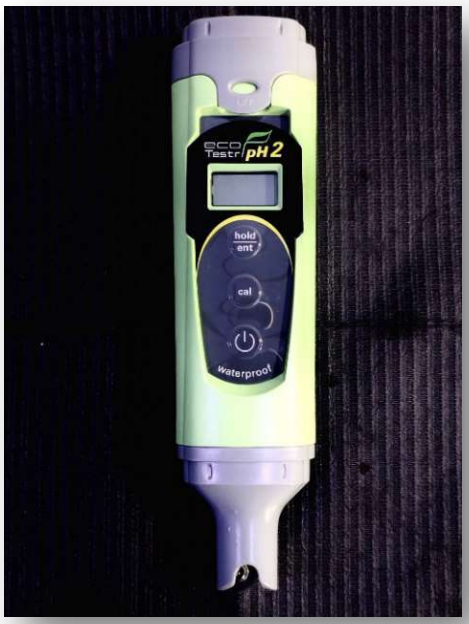

Figure (3.2): Ecotestr PH2 Meter

3.2.3 Sample Collection and Selection:

A total of 108 human mandibular premolars that were freshly extracted for orthodontic purposes were selected from the Department of Orthodontic and Oral Surgery (Center of Mosul) from patients aged 15–25 years and utilized in this study (Figure 3.3).

Figure (3.3): Selected mandibular premolars.

The selected teeth should have straight, fully formed root apices with patent foramina, a single root with type I canal configuration following Weine's classification, and be free from carious lesions (root or coronal). Inclusion criteria involve lower mandibular premolars with a single root and single canal. Exclusion criteria, teeth with dilacerations, anatomical or morphological deformities, cracks or fractures, and immature open apices. The external surface of the roots was cleaned carefully from any remaining soft tissues by a dental scaler, and stored in distilled water in a closed container until used to avoid dehydration. The extracted teeth were radiographed from buccolingual and mesiodistal directions to exclude any teeth with calcifications, resorptive defects (external and/or internal), endodontic treatment, and multiple canals (Figure 3.4). Moreover, a stereomicroscope (10X) was used to exclude any tooth with cracks or defects in the root (Zaki *et al*., 2022; Kadulkar *et al*., 2024).

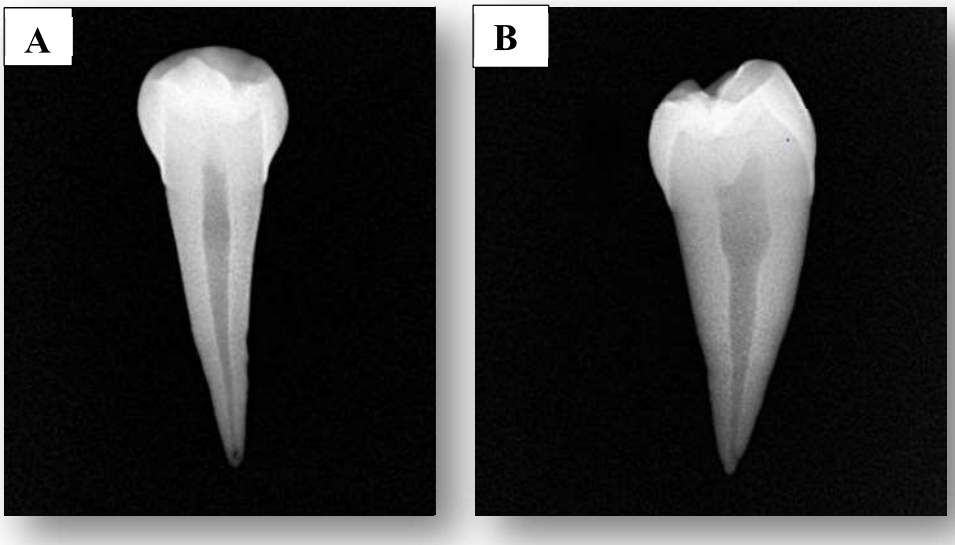

Figure (3.4): (A) Buccolingual periapical radigraph, (B) Mesiodistal periapical radiograph

3.2.4. Tooth mounting and decoronating:

The root length was determined to 12mm by a digital caliper with a pencil dart was used. A Polyvinyl chloride (PVC) tube with 1 inch in diameter and 20 mm in height was used and closed from one side with wax. Silicone rubber dental arch jaw mold of 10 cm in length, 10 cm in width, and 4 cm in height was poured with stone, and 4mm of PVC tube was inserted in the center of the stone before solidifying. After that Polysiloxane elastomer impression material was mixed according to the manufacturer's instructions and filled the PVC tube to construct a custom-made mold. With the help of a dental surveyor, 8 mm of the tooth was mounted in the center of the impression substance before being set as figure (3.5) (Hussien and Al-Gharrawi, 2019).

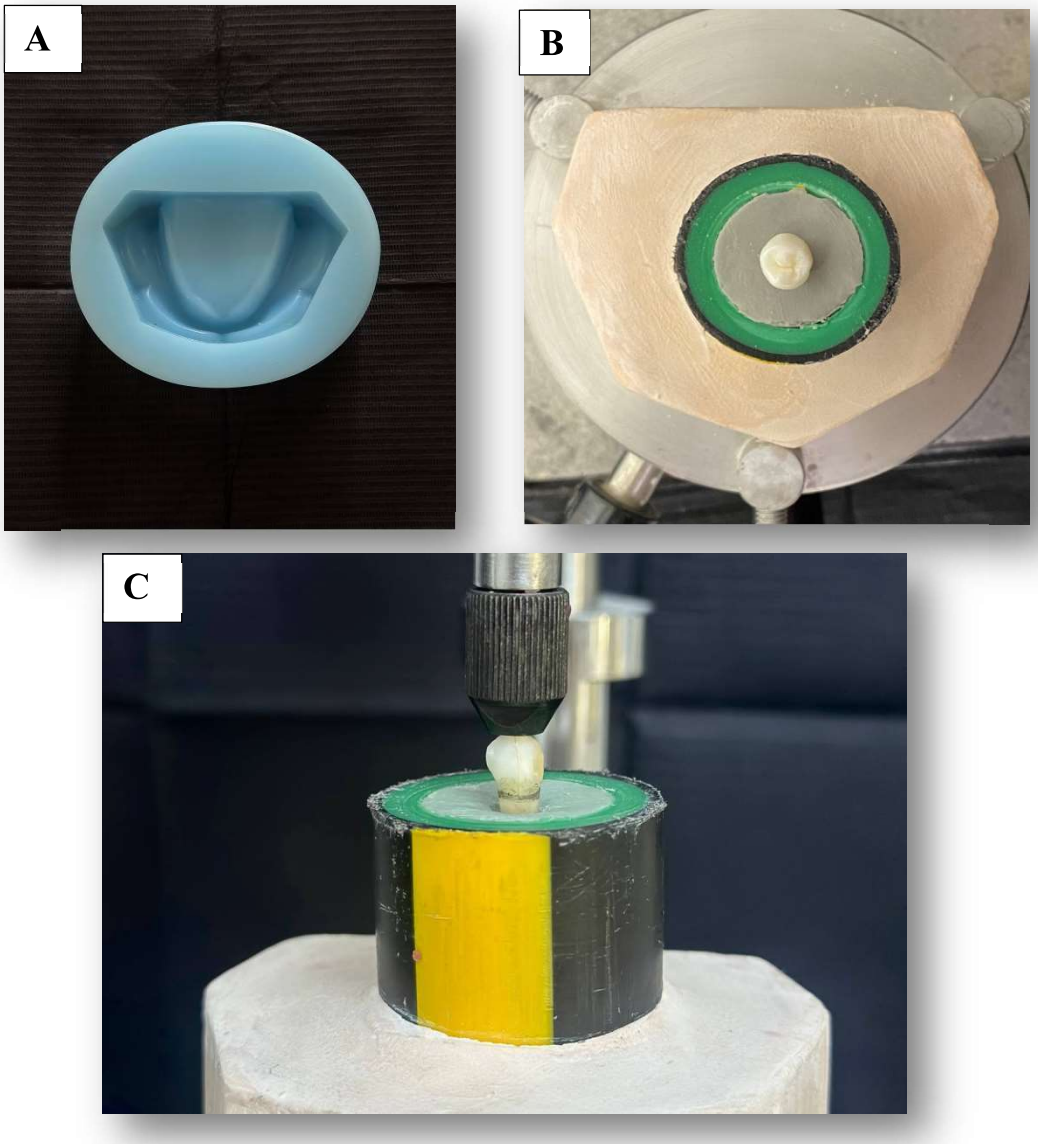

Figure (3.5): Silicone rubber mold (A). Mold poured with stone and PVC inserted in center of stone that filled with polysiloxane elastomer impression material (B). Tooth inserted in the center of impression with the aid of dental surveyor (C).

The method of tooth decoronation was done using an attachment of the air-rotor handpiece to a surveyor. Air-rotor handpiece on the surveyor was oriented by a customized clamp. A protractor was affixed to determine the angulation of the straight pencil dart point (Agrawal *et al*., 2021).

A guiding line was drawn all around the root horizontally with a pencil dart to standardize the root length of the tooth at a length of (12mm) from the anatomical apex and a double-sided diamond disc (0.2mm) in thickness was used to split the tooth horizontally by cutting off coronally perpendicular

to the long axis of the roots under cold water to prevent heating and reduce smearing (Figure 3.6). After a coronal portion of all samples was removed, the length of all samples was confirmed with a digital caliper (Abdullah *et al.*, 2024).

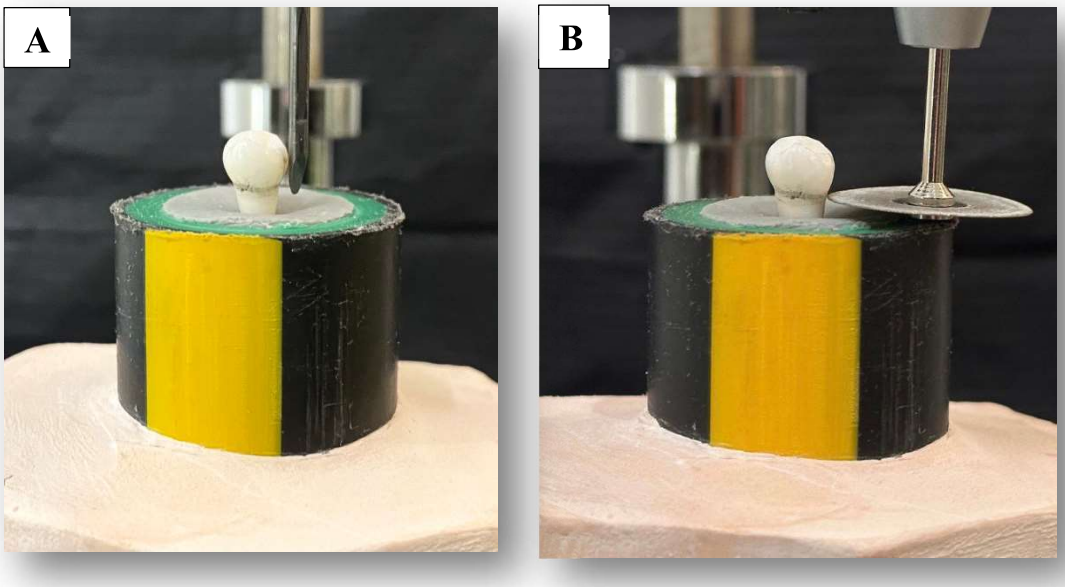

Figure (3.6): (A) Horizontal gidding line with pencil dart, (B) Diamond disc (0.2 m) with slow speed handpiece for cutting coronal portion.

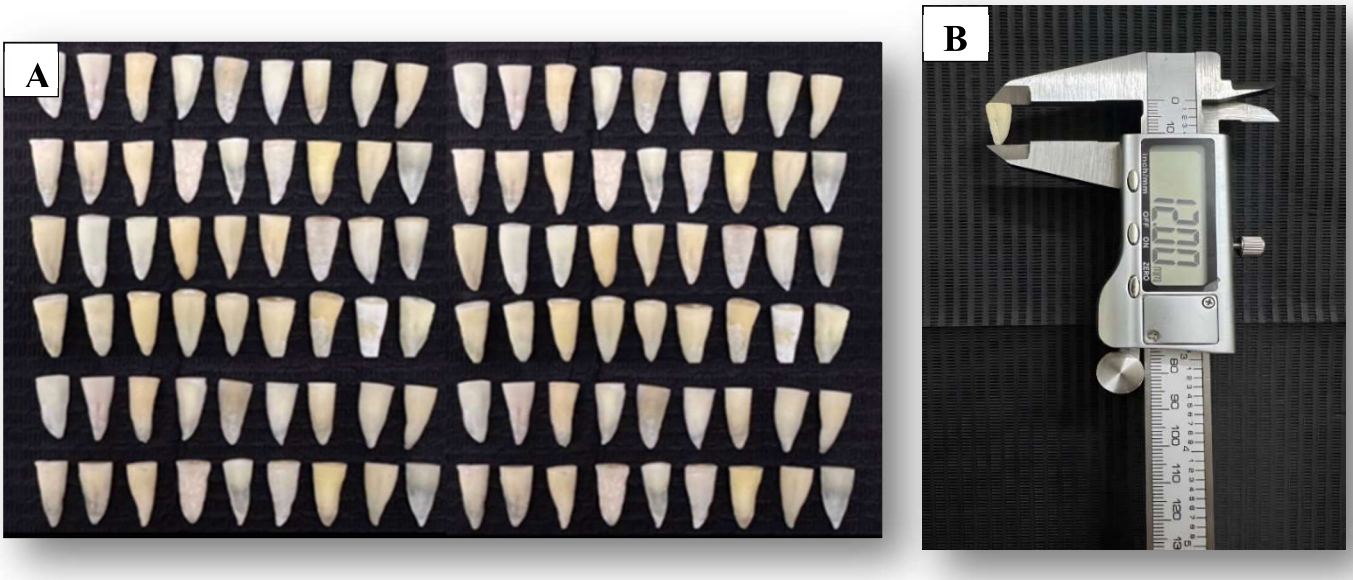

Figure (3.7): (A) The lengths of decoronated teeth. (B) lengths confirmed with digital clipper.

3.2.5. Working length determination:

The roots of decoronated teeth were withdrawn from their simulated sockets. The canals were accessed and pulpal tissues were extracted with a barbed broach. Using the No. 10 K file, the center location of the apical foramen and the exact apical patency of the root canal were confirmed. Next, the correct working length was calculated by deducting (1mm) from the length at which No. 10 K-file was visible at the apical foramen by the necked eye figure (3.8) (Al-Sabawi *et al*., 2012; Ricardo *et al.,* 2020; Majumdar *et al*., 2021).

Figure (3.8): Determination of working length

A closed canal system was prepared. Tray adhesive bond was applied on the exterior surface of each root of decoronated teeth and the roots apex were closed with wax that was allowed to solidify before reinserted in its stimulated socket in impression materials filled PVC tube to be positioned in the surveryor to facilitated canal preparation and obturation. This closed canal system was prepared to prevent extrusion of irrigation and sealer from apical apex but permit recapitulation canal patency figure (3.9) (Raouf and Saeed, 2020; Sadek and Hassan, 2022).

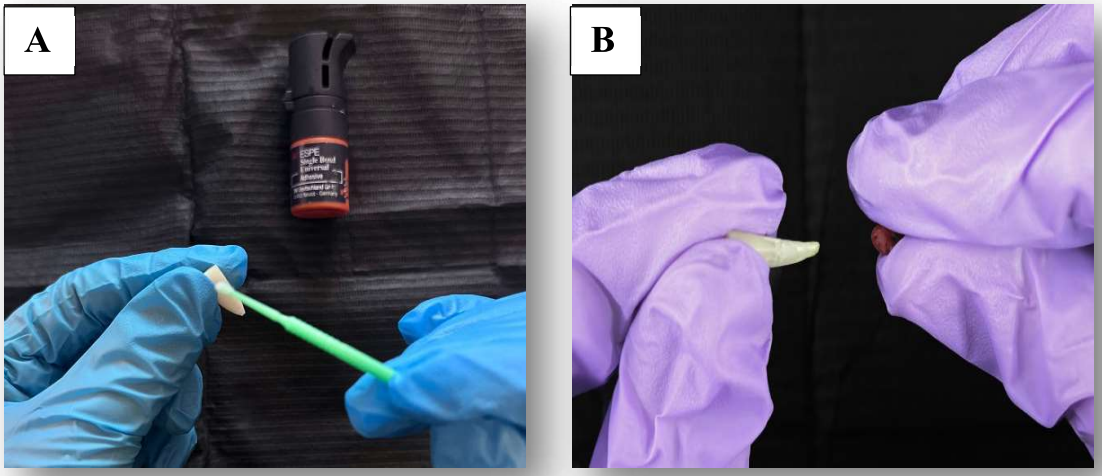

Figure (3.9): Coating root apex of sample with tray adhesive bond (A). Covering the root apex with pink wax (B).

3.2.6. Root Canal Preparation

The preparation of the root canal has been performed with ProTaper universal NiTi rotary files connected to an eighteeth endo-motor with a torque of 3 N/cm and a speed of 250 rpm, according to the manufacturer's instructions. First size, No. 10 K-file was used to construct a reproducible and smooth glide path for all root canals. Then the canals were instrumented with ProTaper universal NiTi rotary files from SX to size F3 following the manufacturer's instructions in a crown-down manner using gentle in and out motion. Irrigation and recapitulation with a size 15 hand file were performed after each file as in figure (3.10) (Antunovic *et al.,* 2021; Majumdar *et al*., 2021).

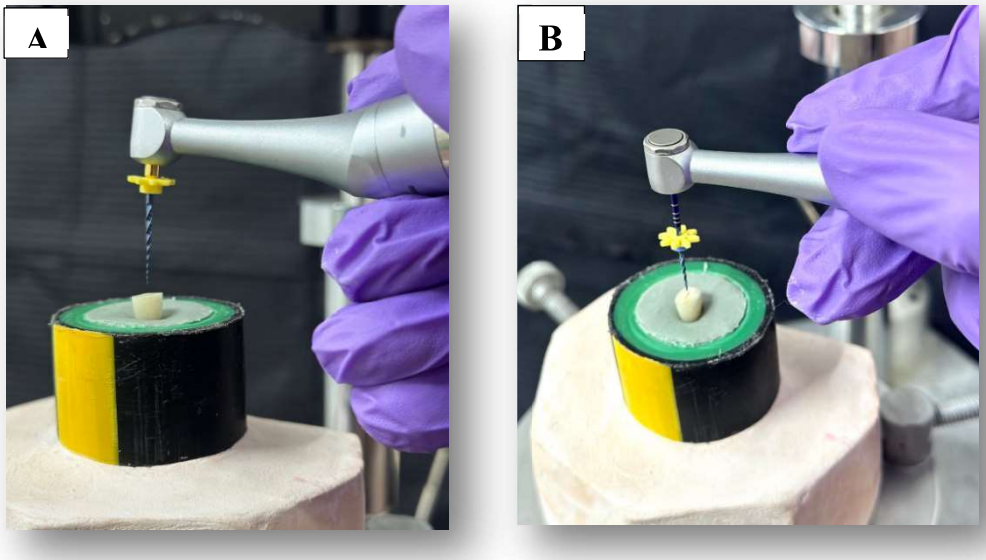

Figure (3.10): Instrumentation of sample with endo-motor (A) Lateral view, (B) Occlusal view.

About 2ml of 2.5% NaOCl solution for (1) min was irrigated in the canals during instrumentation and between each file. A gauge 30 double-sided Vented needle was used and entered 1-2mm short from working length for initial and final irrigation. Finally, root canals were rinsed with 5 mL of distilled water to get rid of irrigation and debris as in figure (3.11) (Hussien and Al-Gharrawi, 2019; Karthi *et al*., 2023).

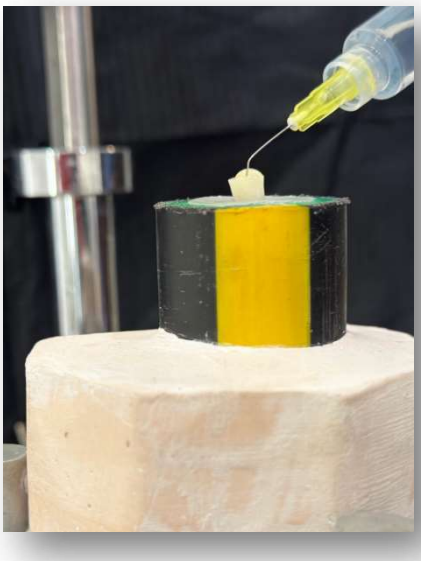

Figure (3.11): Irrigation of sample by disposable endodontic needle.

3.2.7 Sample Distribution

Based on the final irrigation used, five experimental groups and one control group (n =18) were developed and each final irrigant was rinsed the canals about 3 ml for 3 minutes (Ratih *et al*., 2020). The experimental work steps and sample distributions are summarized in (Figure 3.12).

The root canal was filled with 3 mL final irrigants, by using a 3 mL syringe with a 30-G double side-vented needle. The solution remained in the canal for 3 min. Solution was expressed into the root canal by inserting the needle 1 mm short of the working length and moving in an up-and-down motion with an amplitude of 1–2 mm.

The canals were finally washed with (10 ml) of distilled water for 2 min and dried with F3 sterile absorbent paper points (Dhawan *et al*., 2019).

After the root canals had been biomechanically prepared, 9 samples from each group were taken for smear layer removal test under SEM and other 9 samples of each group were utilized for push out bond strength test under universal testing machine.

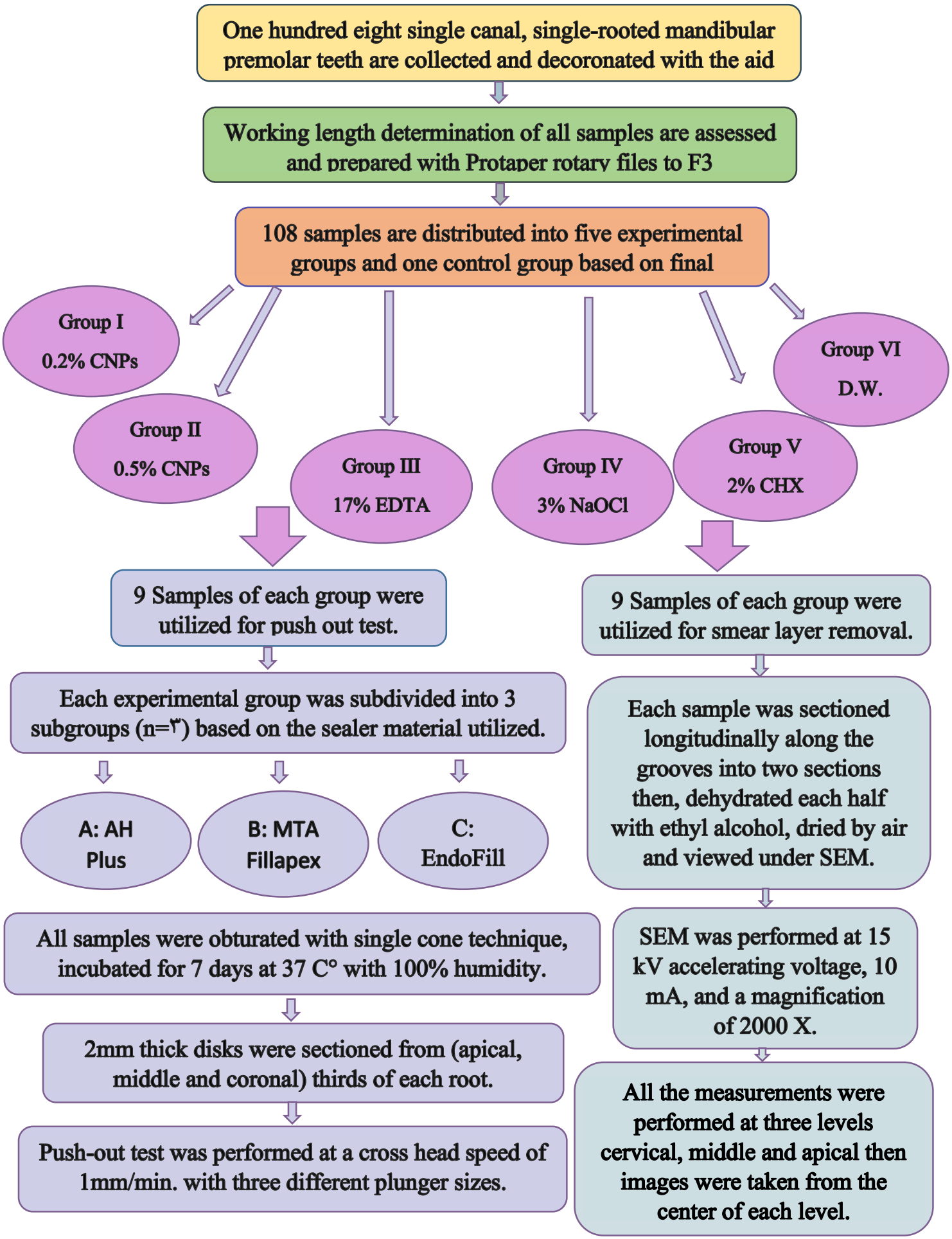

Figure (3.12): Schematic diagram of the experimental work and samples distribution

3.3 Smear Layer Removal

3.3.1. Preparation of Sections for Viewing Under Scanning Electron Microscopy

The fifty- four samples were removed from the mold and fixed them using a bench vice, high-speed diamond disc was used to create two parallel longitudinal grooves without perforating the canal on the buccal and lingual faces of each root. A tiny cotton plug was used to close the canal opening after F3 was inserted inside of it. The gutta-percha cone served as a gauge for the depth of the groove to prevent the disc from penetrating the canals and causing contamination from sectioning-related debris. Next, a chisel was used to split the root longitudinally along the grooves into two sections. All the measurements were performed at three levels: coronal (8– 11 mm from the apex), middle (6–8 mm from the apex), and apical (1–3 mm from the apex) thirds by creating three horizontal grooves using a tapered fissure carbide bur perpendicular to the canal on each half of roots as figure (3.13) showed. Images were carefully taken from the central parts of each region (Shi *et al*., 2023; Oraim and Al-Zaka, 2024).

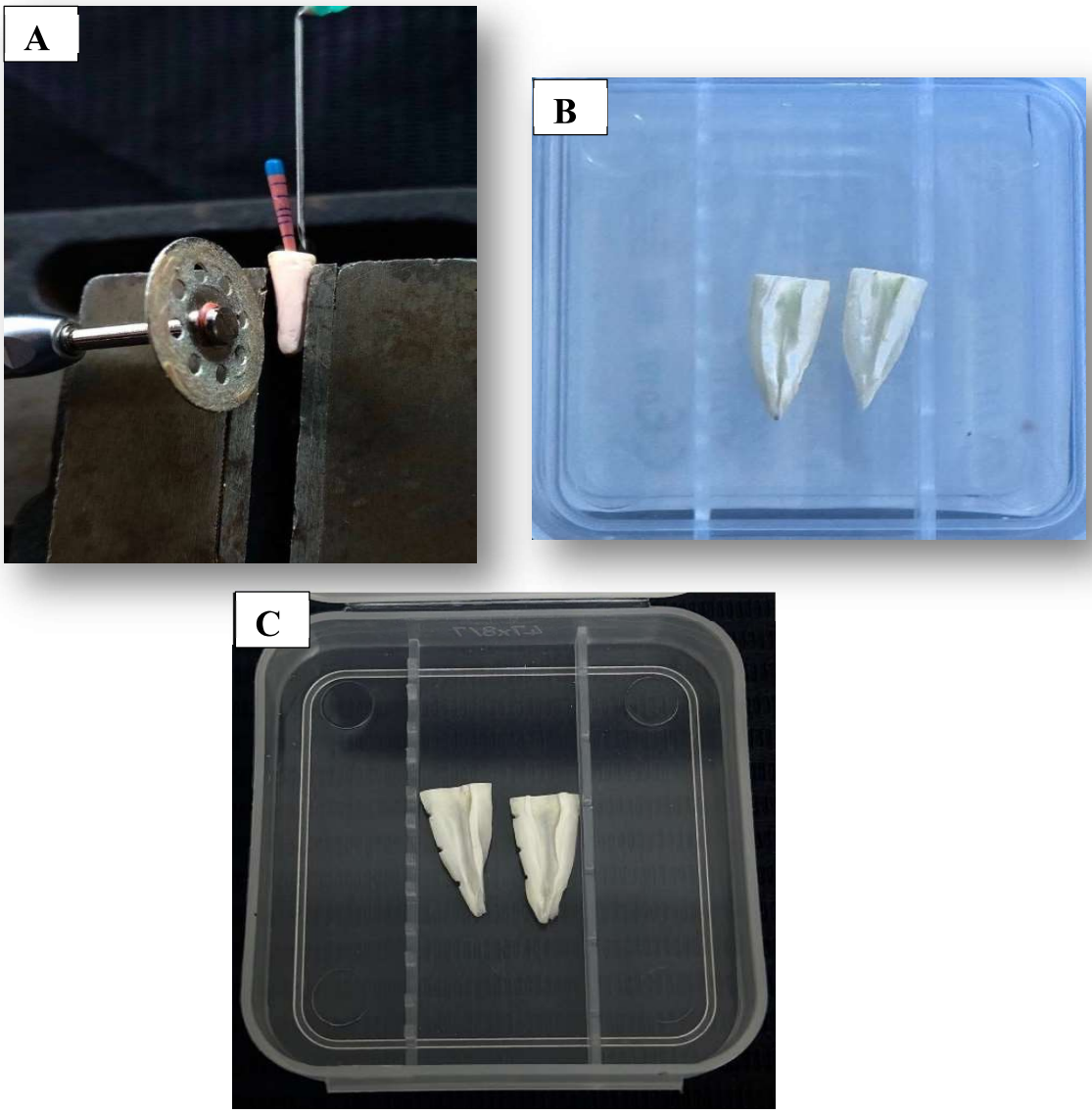

Figure (3.13): Splitting of the tooth, (A) Tooth was fixed by bench vice and parallel longitudinal grooves on the buccal and lingual aspects of root were made by using a high-speed diamond disc under water-cooling. (B) A chisel separates the root into two parts longitudinally along the grooves. (C) Three horizontal grooves on each half of roots.

3.3.2 Scanning Electron Microscopy Examination

Ethyl alcohol 30% for 10 minutes, 50% for 20 minutes, 70% for 20 minutes, 90% for 30 minutes, 100% for 30 minutes, and 100% for 30 minutes was used to dehydrate each half, dried by air. Specimens were sputtered coated with a thin film of gold-palladium (10 nm) under vacuum conditions using a sputter coater (Qurum, Q150R S Plus) to increase electrical

conductivity and reflection of samples and then each specimen was mounted in coded stubs and placed in a vacuum chamber which was then viewed under SEM at 10,13 and 15 kV accelerating voltage, 10 mA, and a magnification of (2000 X). Images were obtained from the central part of each coronal, middle, and apical third of each half root to detect whether the smear layer is present and visualize the opening of tubules that were observed by one observer who is unknown to the irrigation schedules employed for each sample figures (3.14; 3.15; 3.16) (El Hachem *et al*., 2023).

Figure (3.14): Sputter coater (Qurum, Q150R S Plus)

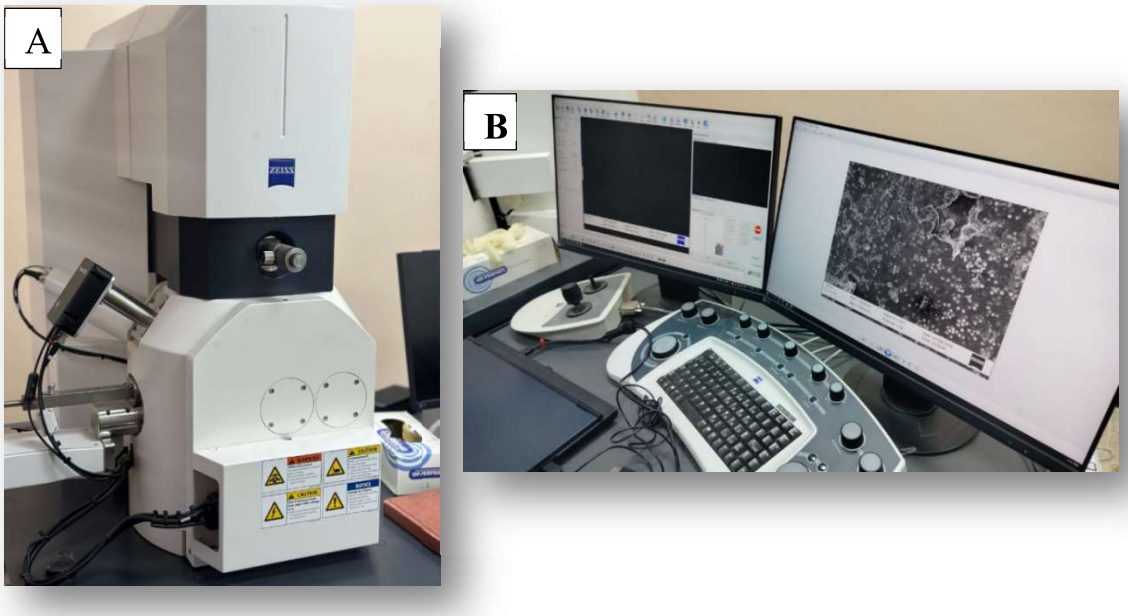

Figure (3.15): (A). Computer system consists of viewing screen to display the scanned images (B). keyboard to control the electron beam

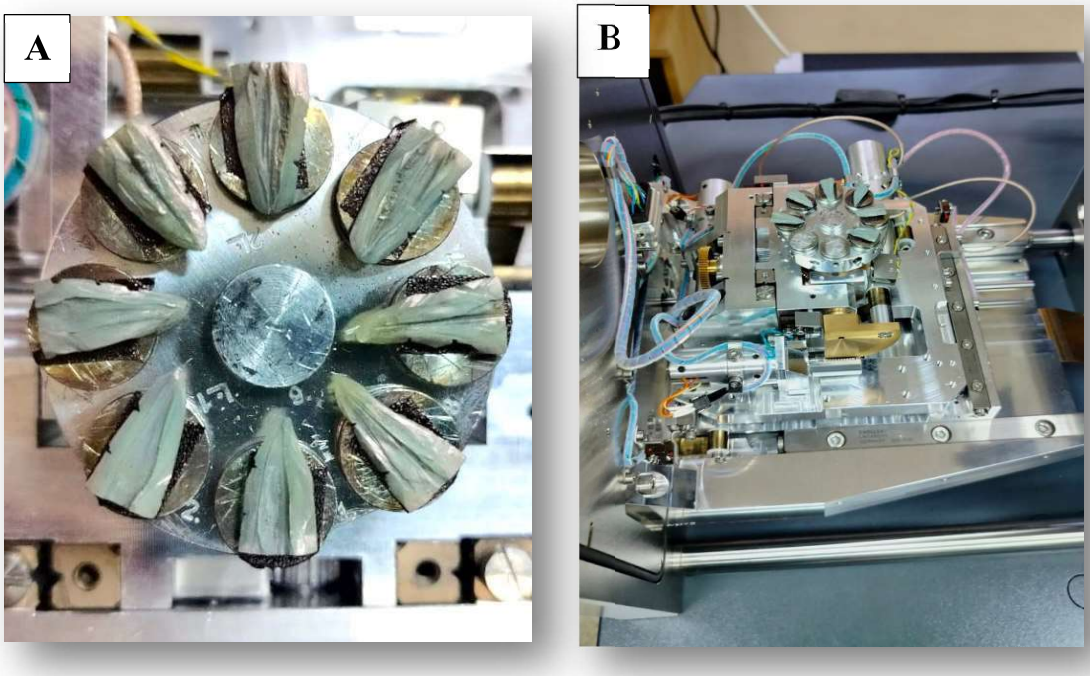

Figure (3.16): (A). Specimens were mounted in stub (B). Specimens placed in the SEM

The images were assessed based on Hülsmann's categorization about being there of smear layer and degree of tubules opening (Çobancı *et al*., 2023):

• Score 1: No smear layer; dentinal tubules are open.

• Score 2: Small amount of smear layer; some dentinal tubules are open.

• Score 3: Homogeneous smear layer covering the root canal wall; only a few dentinal tubules are open.

• Score 4: Homogeneous smear layer covering the root canal wall; no dentinal tubules were open.

• Score 5: Heavy, homogeneous smear layer covering the entire root canal wall; no open dentinal tubules.

3.4 push out bond strength

3.4.1 Root Canal Obturation

The remaining 9 samples have been used for push-out bond strength that had remained in custom-made mold and positioned in the surveyor. Based on the root canal filling sealer, each final irrigation group was randomly divided into three subgroups, each include three samples:

Group A: AH Plus with F3 ProTaper gutta-percha cone.

Group B: MTA Fillapex with F3 ProTaper gutta-percha cone.

Group C: Endo Fill with F3 ProTaper gutta-percha cone.

In all of the groups, root canal obturation was achieved using the single-cone obturation technique. A standardized guttapercha cone of the same size as the master apical file was inserted into the root canal up to the working length.

To achieve tug back to the predefined working length, an F3 ProTaper master cone was first inserted within each canal.

In group (A), AH Plus sealer (Dentsply, Germany) was mixed in uniform volume of both pastes on a mixing pad with plastic spatula according to the manufactures instructions to obtain a homogeneous consistency of the sealer that have been delivered into canal using 30-lentulo spiral (Figure 3.17)

Lentulo spiral was fixed to a contra-angle low speed handpiece that coupled to a micromotor and fastened on a surveyor. The sealer was Covered two-thirds of the lentulo and inserted into the canal at a speed of 300 rpm in a clockwise direction, (1mm) short of the working length. Lentulo spiral standardization was applied three times in all canals for fifteen seconds each. With the handpiece rotating continuously, the lentulo was gradually extracted from the channel. To achieve maximal sealer distribution inside

the canal, the tip of the F3 master cone was also dipped in a thin layer of its respective sealer and introduced into the root canal slowly, moving up and down the pump continously until it reached the necessary working length (Figure 3.18) (El Sayed and Al Husseini, 2018; Ahmed and Al-Mezouri, 2019).

In group (B), MTA Fillapex sealer **(A**ngelus /Brazil) was mixed by using a self-mixing tip attached to a syringe and was delivered into root canals in the same manner of previous sealer.

In group (C), EndoFill sealer (PD, Switzerland) was mixed according to the manufacturer's directions of powder and liquid to form a paste consistency and was delivered into root canals in the same manner of previous sealer.

Excess gutta-percha was removed using a heated plastic instrument and vertical compaction was performed with a plugger 1 mm below the orifice level then the coronal access of all root canals was sealed with a flowable composite barrier and stored in an incubator at 37°C at 100% humidity for 1 week to allow the sealant to solidify and replicate the oral environment (Khullar *et al*., 2021).

Figure (3.17): (A) Sealers were used in the study. (B) Lentulo spiral.

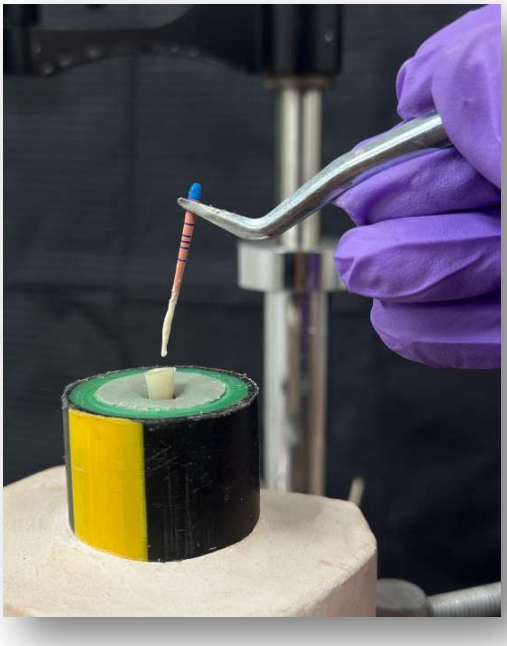

Figure (3.18): F3 Protaper guttapercha was dipped in thin layer of sealer to ensure maximum amount of sealer distribution

3.4.2 Sample Sectioning method:

Each sample was embedded centrally and vertically in a clear cold-cured acrylic resin custom-made mold block with 15mm in length and 10mm in width as in figure (3.19). A waterproof pen was used to mark the acrylic molds as a guide during the root sectioning process and to mark on apical surface of each specimen.

Each root block was fixed with a vice bench to facilitate handling and sectioning. Sectioning was carried out in a horizontal plane perpendicular to the long axis of the root canal with a diamond disc (0.2 mm thickness) at low speed on a straight handpiece with constant fresh cooling water into three equal sections (2mm thickness) where the most apical 3 mm was discarded and the remaining 2 mm apically represented the apical section, then 2 mm thick slice was taken from the middle of the remaining sections to represent middle and coronal root canal thirds. A digital caliper was used to verify the exact thickness of slices as in figure 93.20). Both coronal and apical surfaces were carefully examined to select only a circular root section with a uniform sealer layer to ensure a uniform distribution of the force during push-out

testing and thus accurate measurements. The diameter of the canal in both apical and coronal aspects was measured using a microscope micrometer (ocular micrometer) before subjecting the samples to the push-out test then the sections were kept in 100% humidity till testing (Elmessiry *et al*., 2019; Al-Anazi and Mathew, 2020).

Figure (3.19): Teeth embbed in clear cold cure resin block

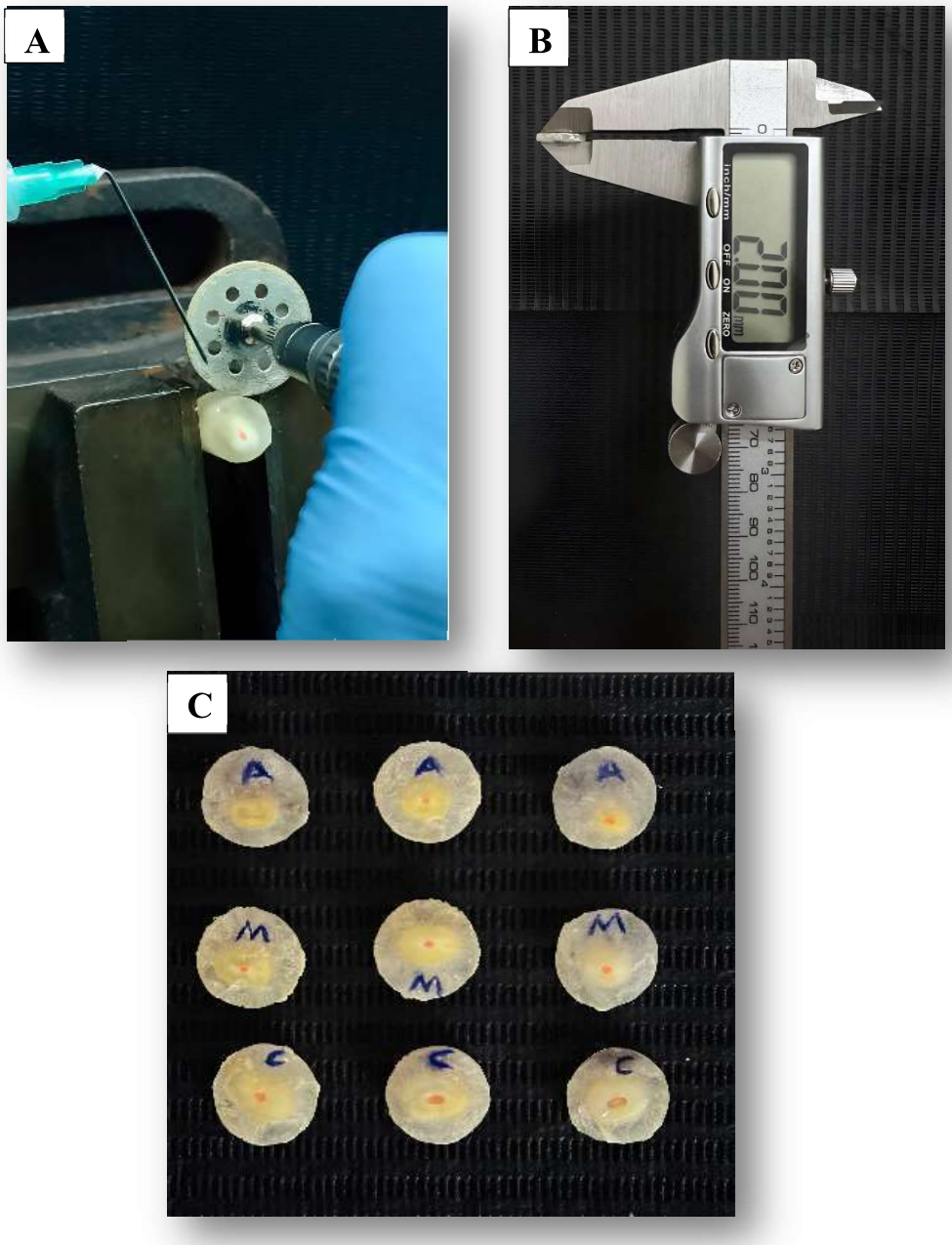

Figure (3.20): (A) Tooth mold secured in a vice bench and root sectioned using a diamond disc, (B) verification of slice thickness using electronic digital caliper, and (C) the resultant root slices

3.4.3 Push out bond strength application test Evaluation

Each section was positioned on a metal base with a central hole with an apical surface of the section that had a smaller diameter facing the plunger. The center of the tested specimen was aligned over the hole so that the filling material could fall freely through once the bond between the dentin wall and the test material was broken (Figure 3.21) (Majeed and AlShwaimi, 2017; Attash and AL-Ashou, 2022).

Cylindrical stainless-steel plunger with diameters 0.7,0.5 and 0.3 mm corresponding to coronal, middle, and apical sections respectively used to load root filling of each section. The tip of the plunger was adjusted to cover the root-filling material without touching the canal wall. The plunger was mounted to a computer software-managed universal testing machine and applied load was applied in an apical to the coronal direction to avoid any impediments during the push-out testing because of tapering of the root canal. A cross-head speed of 1 mm/min was conducted until the first dislodgment of obturating material and a sudden drop along the load-deflection and the maximum force conducted for debonding was measured in Newton (N). The bond strength of the sealer was measured in megapascal MPa and calculated by dividing the maximum debonding force measured in newtons (N) over surface area adhesion (A)(mm2) (Naeem and Al-Zaka, 2021; Salem *et al*., 2023; Hassoon *et al*., 2023).

Bond Strength (MPa)=debonding force (N) /Surface area (mm2)(A)

$$\mathbf{A(mm^2) = \pi(r_1 + r_2)\sqrt{[(r_1 - r_2)^2 + h^2]}}$$

h= Thickness of section in mm, π= 3.14, r_1 is the coronal radius, r_2 is the apical radius (Naeem and Al-Zaka, 2021).

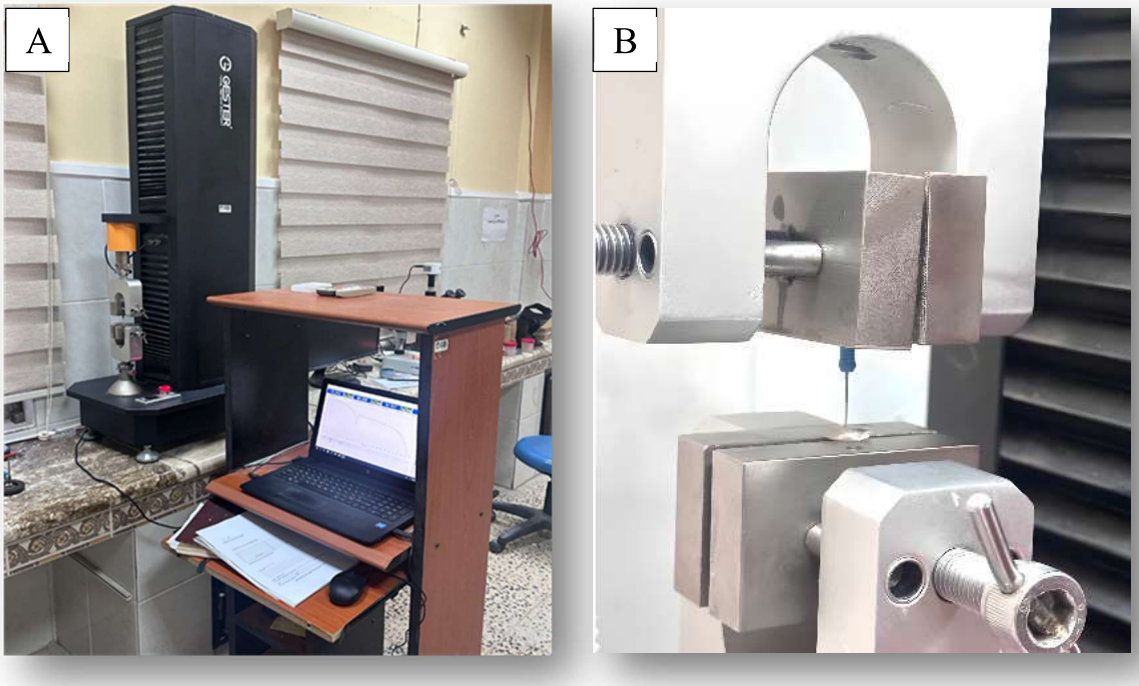

Figure (3.21): (A) Universal testing machine, (B) The sample centered under the plunger with metal base

3.5 Mode of failures assessment:

The mode of bond failure after the push-out test was observed under a stereomicroscope at (20X) magnification (Figure 3.21). The failures were categorized into three types: type I (adhesive failure at the sealer-dentin interface, no residual material on the canal wall), type II (cohesive failure within the obturating material, material exists on entire canal wall), or type III (mixed failure in both sealer and dentin, material present as patches on canal wall) (Al-Askary *et al*., 2013; Donnermeyer *et al*., 2019; Özata *et al.,* 2023).

Figure (3.21): Optika, Italy stereomicroscope

3.6 Statistical analysis:

All collected data was subjected to statistical analysis by the Statistical Package for the Social Sciences (SPSS) software, version 24, for Windows (IBM, Armonk, New York).

1. The normal distribution of data was checked, and then the types of tests were selected as a result.

2. Descriptive statistics, which include median, mean rank, maximum, minimum, and standard deviation values for the smear layer removal test. Mean, standard deviation, standard error, minimum, and maximum for push-out test.
3. Kruskal-Wallis test and Mann-Whitney test were conducted in order to find if there are significant differences between different irrigations solutions groups at three thirds regions.
4. Dunn's multiple range test was used to detect the site of significance.

For all previous tests, when the level of statistical significance $p \leq 0.05$, it was considered statistically significant; when $p > 0.05$, it was considered statistically not significant; and when $p < 0.01$, it was considered statistically highly significant.

The Shapiro-Wilk test was used to test normality, the data in the SL removal test was found to be abnormally distributed, and therefore, nonparametric statistical tests were conducted.

CHAPTER FOUR

RESULTS

CHAPTER FOUR

RESULTS

4.1 Results of Smear Layer Removal

4.1.1 Comparison Within Each Group at Three Levels of Root Canal

Kruskal-Wallis analysis showed highly significant statistical difference in the smear layer removal within G I and G VI (P<0.01), and showed significant statistical difference within G II (p<0.05), but showed no significant statistical difference among thirds of GIII, GIV and GV. Dunn's test showed the presence of a significant difference between apical third and coronal third of GI and a significant difference between apical third and coronal middle thirds of GII and GVI (Table 4.1).

Table (4.1): Intergroup comparison and frequencies of SL removal score

Groups		N	MR	St.devation	P _value	Smear layer removal score				
						1	2	3	4	5
G I	Apical	9	20.56[a]	1.00000	0.002 **		11%	34%	33%	22%
	Middle	9	13.06[ab]	0.52705			45%	56%		
	Coronal	9	8.39[b]	0.70711		22%	56%	22%		
G II	Apical	9	20.06[a]	1.00000	0.01*		11%	34%	33%	22%
	Middle	9	10.67[b]	0.52705			56%	45%		
	Coronal	9	11.28[b]	0.72648		11%	33%	56%		
G III	Apical	9	14.61[a]	1.61589	0.85	22%	11%	34%		33%
	Middle	9	14.56[a]	1.11803		11%	11%	56%	11%	11%
	Coronal	9	12.83[a]	1.09291		11%	22%	56%		11%
G IV	Apical	9	14.06[a]	1.22474	0.06		45%	33%		22%
	Middle	9	9.83[a]	0.50000			67%	33%		
	Coronal	9	18.11[a]	1.13039			22%	22%	33%	22%
G V	Apical	9	12.06[a]	1.39443	0.097		45%	22%		33%
	Middle	9	11.44[a]	0.78174			22%	45%	33%	
	Coronal	9	18.50[a]	0.78174				22%	45%	33%
G VI	Apical	9	21.00[a]	0.00000	0.00**					100%
	Middle	9	10.50[b]	0.70711				22%	56%	22%
	Coronal	9	10.50[b]	0.70711				22%	56%	22%

N: number of samples, MR: mean rank, St: standard devation.

** statistically highly significant difference (P<0.01)

* statistically significant difference (P<0.05)

4.1.2 Comparison Between 0.2% CNP and 0.5% CNP Groups with Other Groups at Three Levels of Root Canal

Kruskal Wallis analysis exhibited highly significant difference values for removal of sl among test groups ($P<0.01$) which can be explained in table (4.2, 4.3). The Post hoc test showed that Group I and II were significantly difference from Group VI at middle and coronal thirds ($P<0.05$) and there was no difference among other groups at three sections of tooth ($p>0.05$). Group I was significantly difference with Group V at coronal third.

Group I (MR: 20.17) and Group II (MR: 20.17) showed the highest SL removal among the test groups at apical third although no statistically difference from Group III, VI, V. while Group III (MR: 27.67; 26.66) showed the least ability to remove SL at apical third than Group I, II, VI. Group IV at coronal (MR: 17.72; 15.94) and middle (MR: 13.33; 13.83) thirds showed higher SL removal than Group I and II in sequence although no statistical difference between them. Group V (MR: 44.00; 36.22; 30.94) from Group I, (MR: 4.00;36.66; 30.61) from Group II at apical, middle and coronal and Group VI (MR: 30.78; 25.11; 32.06) from Group I, (MR: 36.33; 25.44; 31.72) from Group II at apical, middle and coronal in sequence showed the least ability to eliminate SL among test groups.

Table (4.2): Kruskal Wallis analysis for SL removal scores between (GI and other study groups) in each tooth section

Groups		N	MR	P-value
Apical	0.2% CNP (G I)	9	20.17^{ab}	0.01**
	17% EDTA (G III)	9	27.67^{a}	
	3% NaOCl (G IV)	9	22.72^{a}	
	2% CHX (G V)	9	30.78^{a}	
	Distilled water (G VI)	9	44.00^{b}	
Middle	0.2% CNP (G I)	9	16.89^{a}	0.001**
	17% EDTA (G III)	9	16.44^{ab}	
	3% NaOCl (G IV)	9	13.33^{a}	
	2% CHX (G V)	9	25.11^{a}	
	Distilled water (G VI)	9	36.22^{b}	
Coronal	0.2% CNP (G I)	9	19.11^{a}	0.00**
	17% EDTA (G III)	9	19.17^{ab}	
	3% NaOCl (G IV)	9	17.72^{ab}	
	2% CHX (G V)	9	32.06^{b}	
	Distilled water (G VI)	9	30.94^{b}	

P<0.01, N: number of samples; MR: mean rank.

Table (4.3): Kruskal Wallis analysis for SL removal scores between (GII and other study groups) in each section of tooth.

Groups		N	MR	P-value
Apical	0.5% CNP (G II)	9	20.17^{ab}	0.01**
	17% EDTA (G III)	9	26.66^{a}	
	3% NaOCl (G IV)	9	23.33^{a}	
	2% CHX (G V)	9	36.33^{a}	
	Distilled water (G VI)	9	44.00^{b}	
Middle	0.5% CNP (G II)	9	15.61^{a}	0.001**
	17% EDTA (G III)	9	15.78^{ab}	
	3% NaOCl (G IV)	9	13.83^{a}	
	2% CHX (G V)	9	25.44^{ab}	
	Distilled water (G VI)	9	36.33^{b}	
Coronal	0.5% CNP (G II)	9	17.89^{a}	0.001**
	17% EDTA (G III)	9	17.83^{ab}	
	3% NaOCl (G IV)	9	15.94^{ab}	
	2% CHX (G V)	9	31.72^{ab}	
	Distilled water (G VI)	9	30.61^{b}	

P<0.01, N: number of samples; MR: mean rank.

4.1.3 Comparison Between 0.2% and 0.5% CNP Groups

Mann-Whitney analysis was done for intergroup comparing between 0.2% and 0.5% CNPs and showed no statistically difference (Table 4.4).

Table (4.4): Mann-Whitney test for indicating the significantly difference in removal of SL for GI and GII at three third regions.

Site	Groups	N	Mann-Whitney U	P- value	Sig.
Apical	G I	9	40.50	1.00	Not Sig
	G II	9			
Middle	G I	9	36.00	0.73	Not Sig
	G II	9			
Coronal	G I	9	26.50	0.22	Not Sig
	G II	9			

p>0.05, Sig: Significance.

GV and GVI show higher SL and debris at all levels of tooth. GI and GII showed better eliminating SL at apical level than cervical and middle levels. GIII showed less eliminating smear at apical third than GI and GII. GI, GII and GIII showed comparable effect in removing SL and less debris was present. GIV exhibited better removal SL at cervical and middle levels as shown in figures (4.1, 4.2, 4.3, 4.4, 4.5, 4.6)

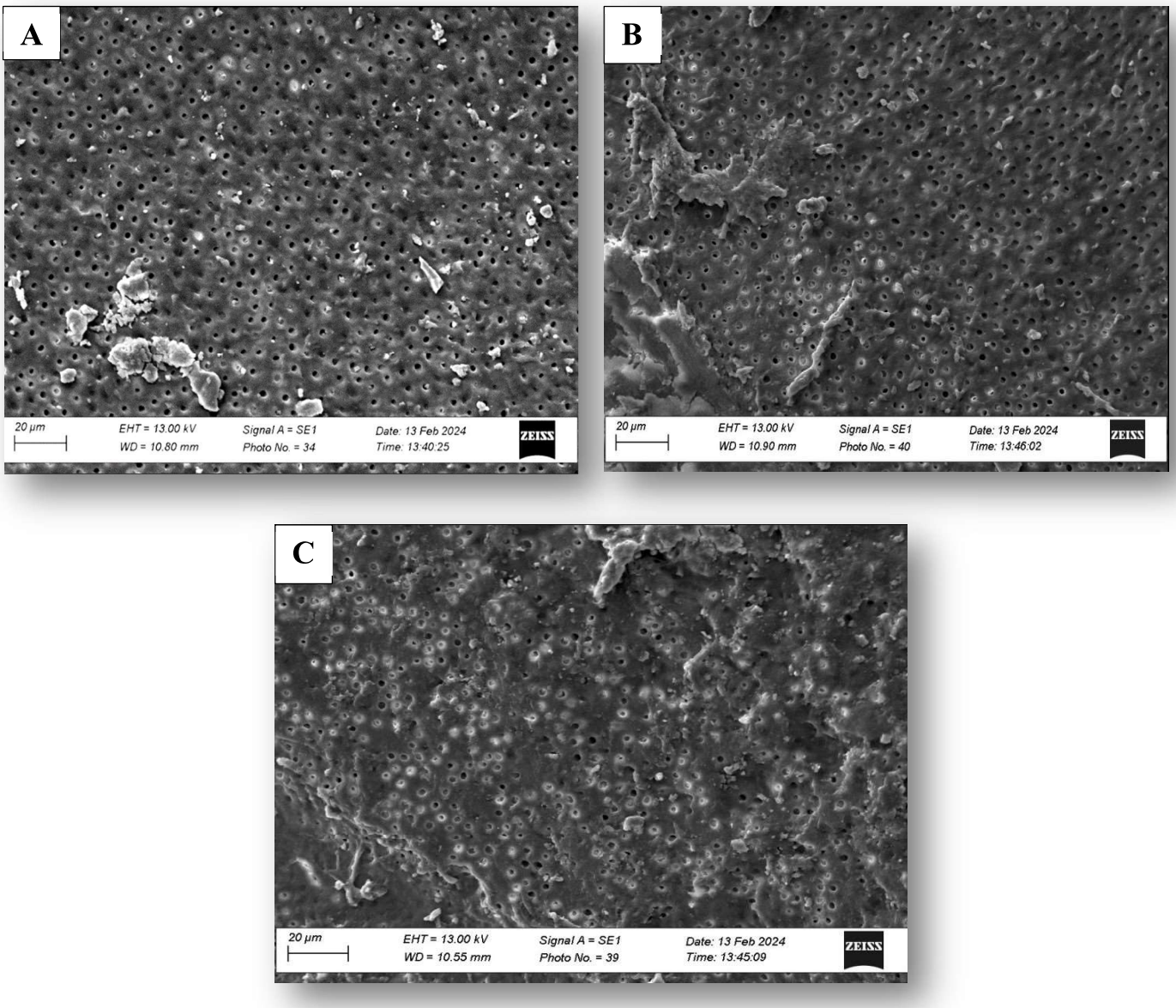

Figure (4.1): SEM photomicrograph of GI showing better eliminating smear layer at (A) apical level than (C) cervical and (B) middle levels.

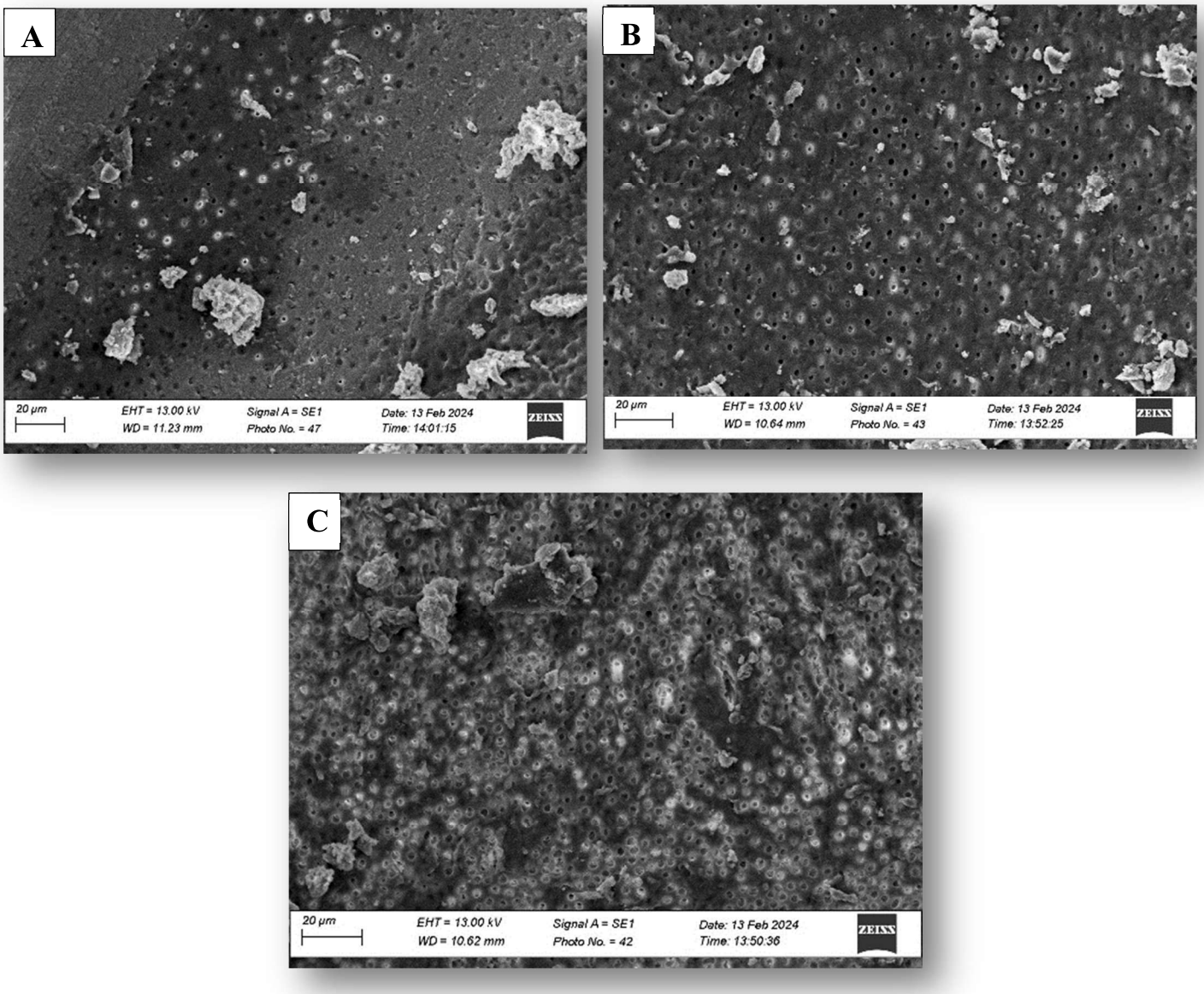

Figure (4.2): SEM photomicrograph of GII showing better eliminating smear layer at (A) apical level than (C) cervical and (B) middle levels.

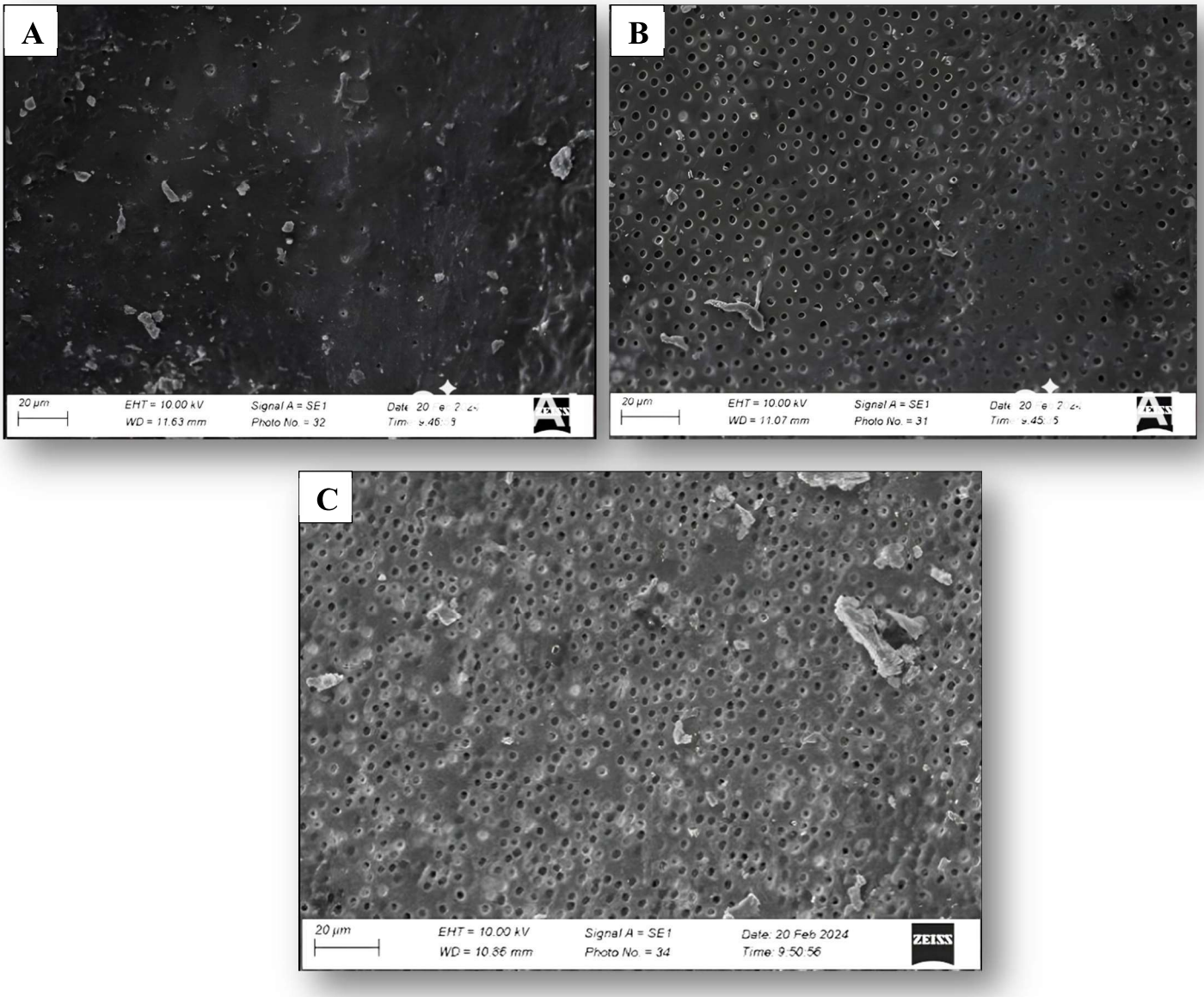

Figure (4.3): SEM photomicrograph of GIII showing less eliminating smear layer at (A) apical third than GI and GII, and comparable effect in removing smear layer and less debris was present at (B) middle and (C) cervical levels to GI and GII.

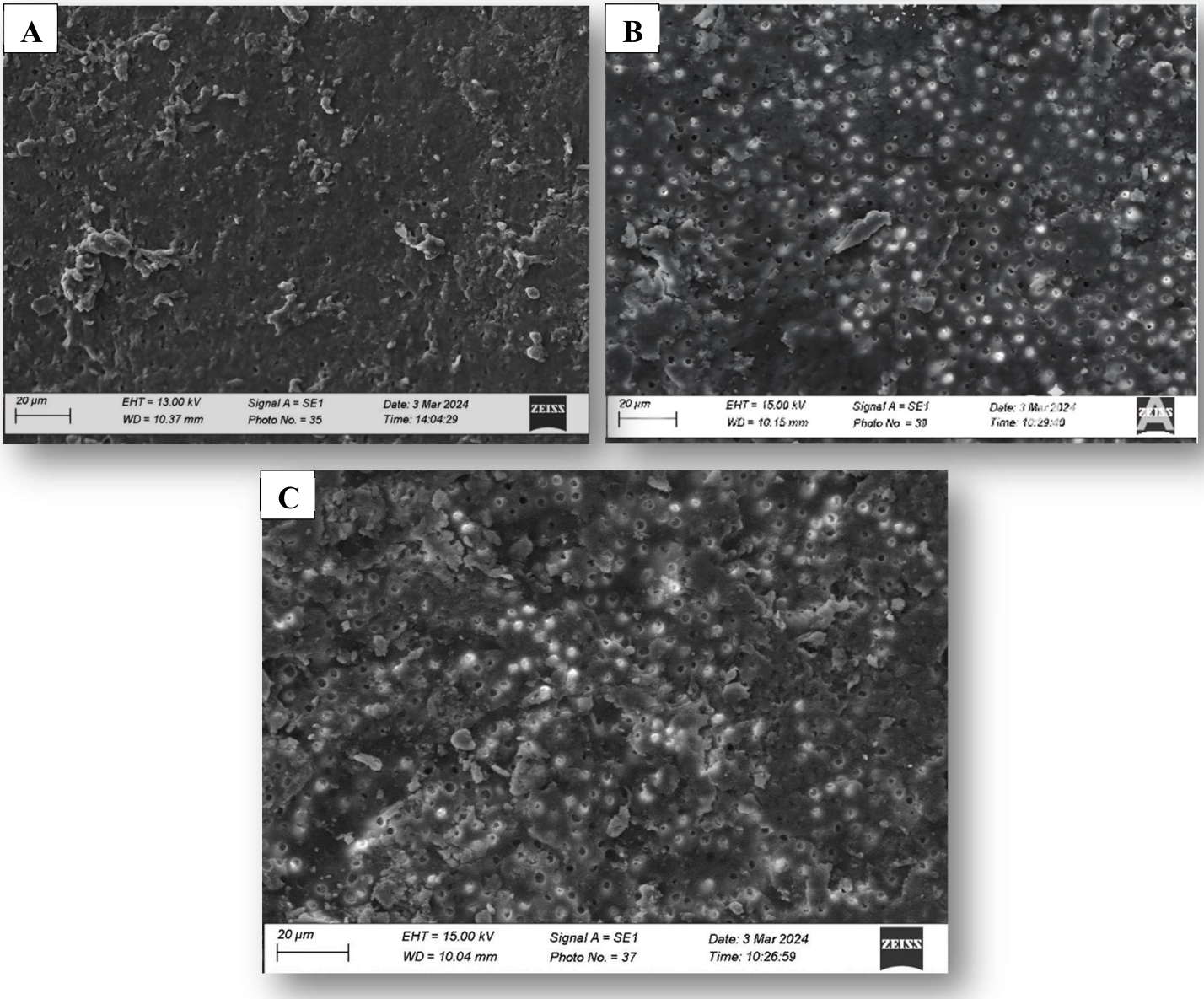

Figure (4.4): SEM photomicrograph of GIV showing less eliminating smear layer and opening of dentinal tubules at (A) Apical level than (B) Middle and (C) Cervical.

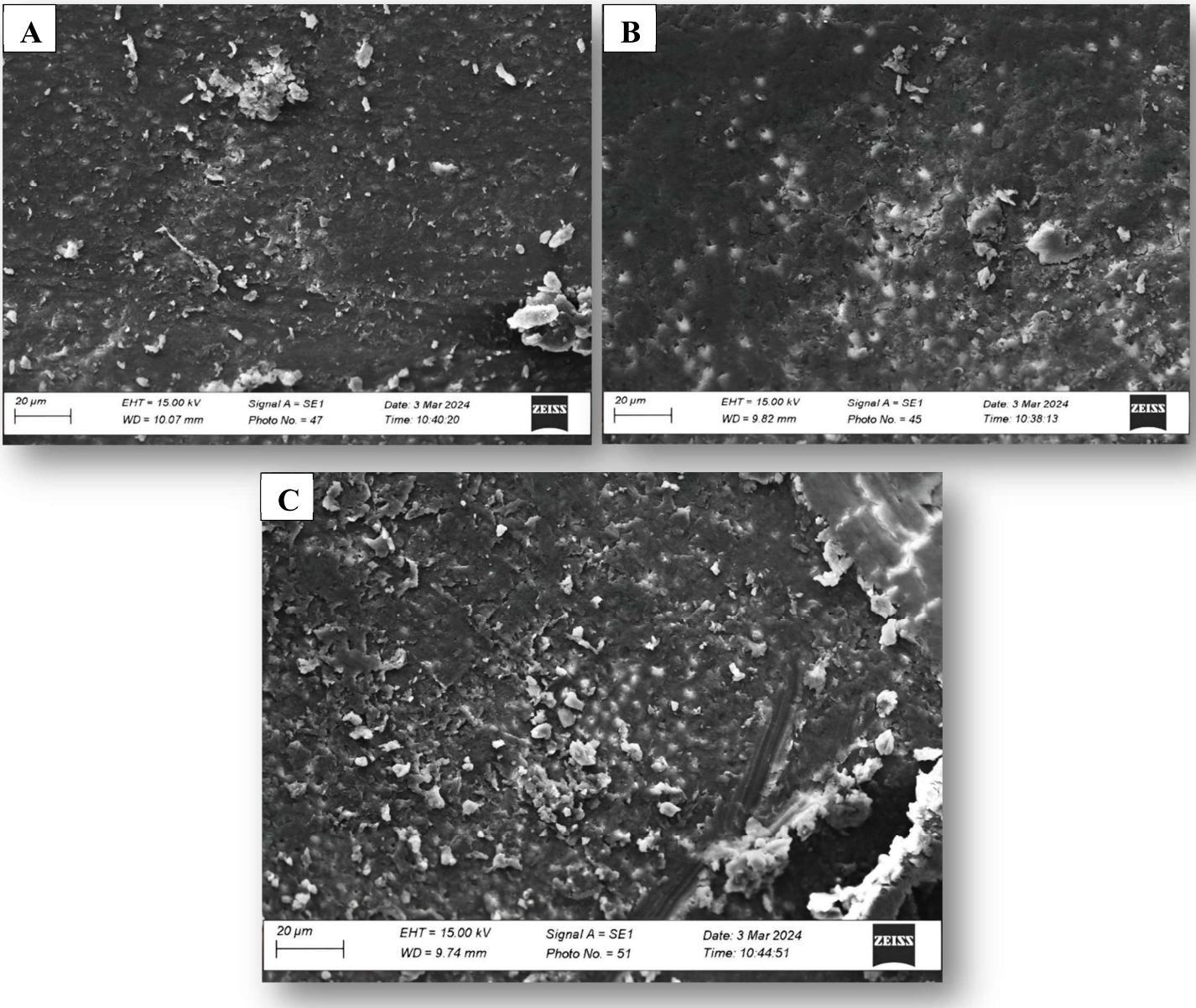

Figure (4.5): SEM photomicrograph GV showing higher smear layer and debris and no opening of dentinal tubules at all levels of tooth, (A) Apical level, (B) Middle and (C) Cervical.

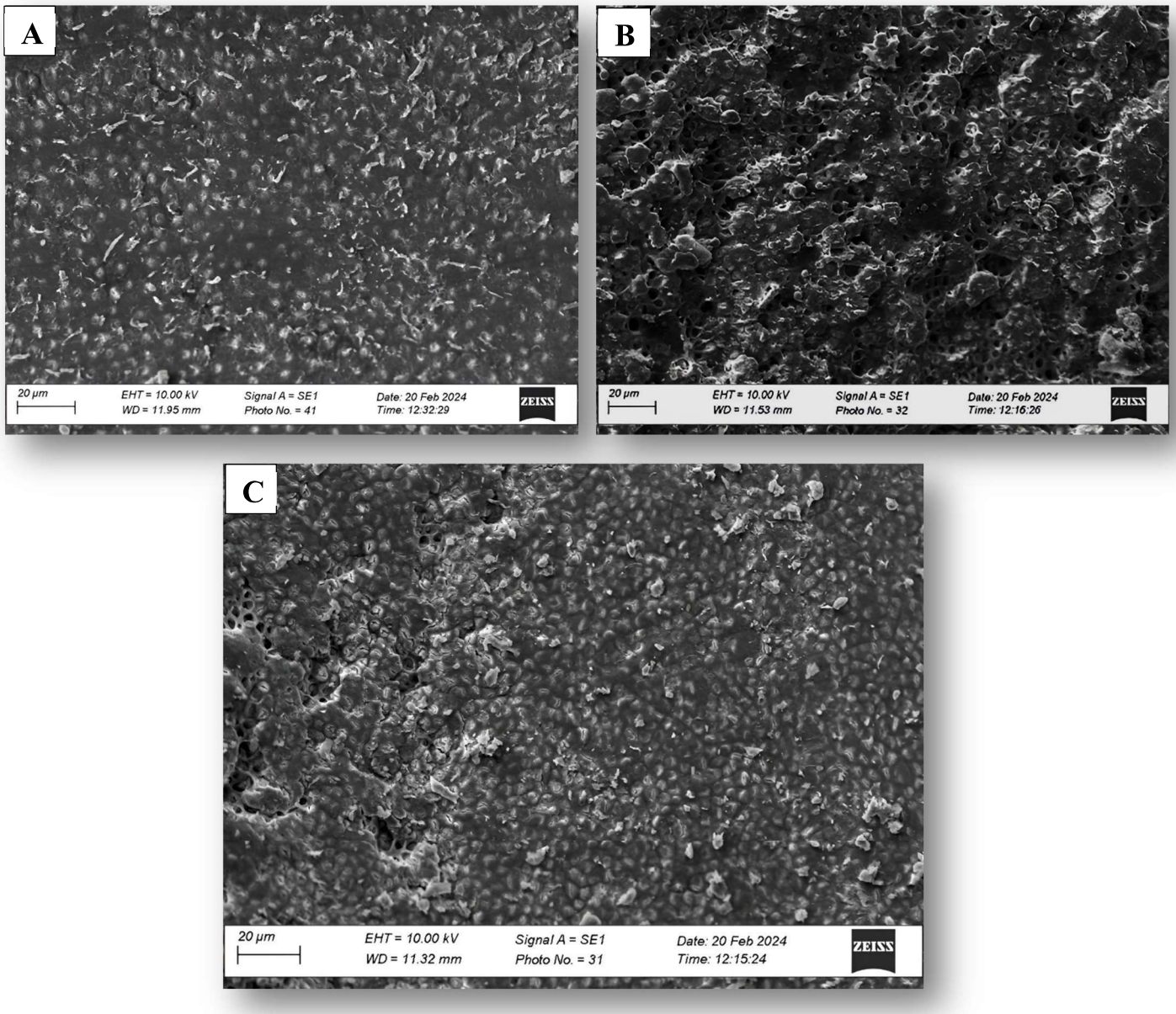

Figure (4.6): SEM photomicrograph GVI showing higher smear layer and debris at all levels of tooth, (A) Apical level, (B) Middle and (C) Cervical.

In figure (4.7), A (Apical), GI and GII, 34% of the score are 3. In G III, 34% of the score are 3. In GIV and GV, 45% of the score are 2. In GVI (Distilled water control group) shows that all of the samples were scored as 5. B (Middle), GI and GIII, 56% of the score are 3. but in GII are 2. In G IV, 67% of the score are 2. In GV, 45% of the score are 3 and 33% are 4. In GVI, 56% are 4. C (Cervical), GI, 56% of the score are 2 but in GII and G III are 3. In G IV, 34% of the score are 4. In GV, 45% of the score are 4. In G VI, 56% of the score are 4.

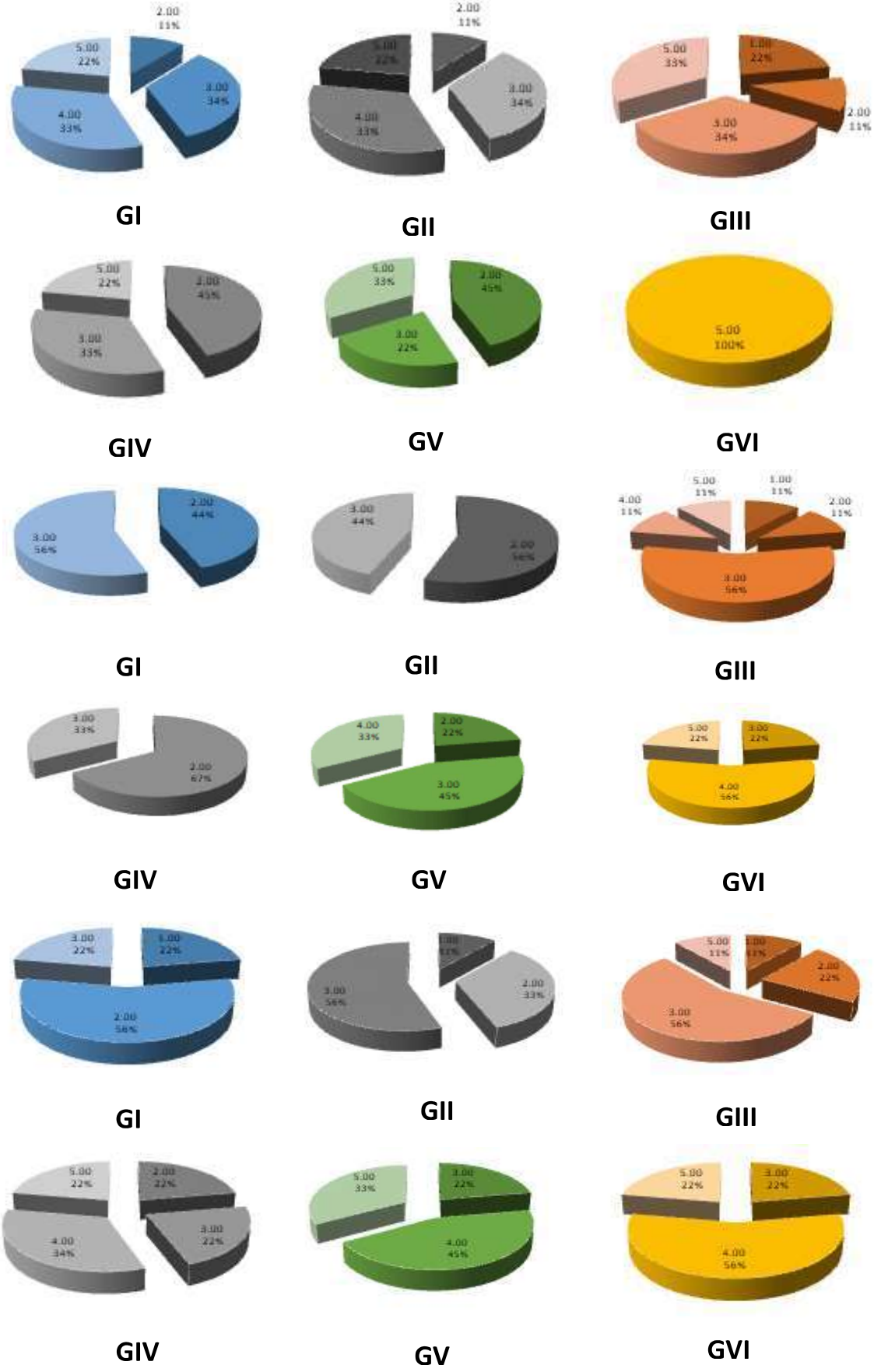

Figure (4.7): A, B and C: Pie chart of scores in three levels for GI (0.2% Nano Chitosan), GII (0.5% Nano Chitosan), GIII (17% EDTA), GIV (3% NaOCl), GV (2% CHX), and GVI (Distilled water)

4.2 Results of Push Out Bond Strength

4.2.1 Descriptive Statistics

The descriptive statistics (mean ± standard deviation) of push-out bond strength values for each sealer subgroups (A, B, C) in different root thirds (apical, middle and coronal) which were irrigated with six groups are shown in table (4.5; 4.6; 4.7) figure (4.8). In GI showed higher bond strength mean values (11.79 ± 9.27) of group (A), (12.43 ± 11.74) of group (B) and (9.8 ± 13.82) of group (C) at apical level than middle and coronal levels. GII showed higher mean values at apical level than mid and coronal level of subgroup A, B, and C (13.79 ± 8.26), (9.41 ± 4.96) and (9.41 ± 4.96) respectively. GIII showed higher mean value at apical level than middle and coronal of group (A) (5.54 ± 4.13), group (B) (5.72 ± 2.57) and group (C) (4.76 ± 2.96). The chelating agents 0.2, 0.5 CNPs have positive effected on push out bond strength of all sealers than 17% EDTA. GV showed higher mean push out bond strength value (9.63 ± 14.03) at coronal of group (A), at middle third (9 ± 12.23) of group (B) and apical third (9.34 ± 11.89) of group (C). CHX improved dislodgement resistance of AH Plus and MTA Fillapex sealer. GIV higher mean (5.42 ± 4.95) at apical than middle and coronal levels of A. NaOCl have negative effected on push out of AH Plus.

Table (4.5): Descriptive statistics (mean and standard deviation) for three tested sealers in six groups at apical third

Groups	AH Plus	MTA Fillapex	EndoFill
	Mean (MPa) ± SD.	Mean (MPa) ± SD.	Mean (MPa) ± SD.
Group I	11.79 ± 9.27	12.43 ± 11.74	9.8 ± 13.82
Group II	13.79 ± 8.26	9.41 ± 4.96	9.41 ± 4.96
Group III	5.54 ± 4.13	5.72 ± 2.57	4.76 ± 2.96
Group IV	5.42 ± 4.95	3.87 ± 3.58	4.31 ± 2.9
Group V	3.18 ± 4.7	3.95 ± 3.41	9.34 ± 11.89
Group VI	5.83 ± 2.7	6.07 ± 3.12	6.56 ± 2.67

SD: Standard deviation, MPa: Megapascale

Table (4.6): Descriptive statistics (mean and standard deviation) for three tested sealers in six groups at middle third

Groups	AH Plus	MTA Fillapex	EndoFill
	Mean (MPa) ± SD.	Mean (MPa) ± SD.	Mean (MPa) ± SD.
Group I	11.62 ± 11.34	7.33 ± 3.45	9.32 ± 3.93
Group II	13.67 ± 4.89	7.33 ± 3.45	9.17 ± 11
Group III	4.6 ± 3.77	4.65 ± 4.98	4.57 ± 5.09
Group IV	3.85 ± 3.54	1.13 ± 1.22	1.11 ± 1.24
Group V	1.43 ± 1.86	9 ± 12.233	4.99 ± 1.8
Group VI	2.08 ± 0.87	3.48 ± 3.22	3.2 ± 3.53

SD: Standard deviation, MPa: Megapascale

Table (4.7): Descriptive statistics (mean and standard deviation) for three tested sealers in six groups at cervical third

Groups	AH Plus	MTA Fillapex	EndoFill
	Mean (MPa) ± SD.	Mean (MPa) ± SD.	Mean (MPa) ± SD.
Group I	3.39 ± 4.3	3.14 ± 3.89	6.86 ± 6.44
Group II	3.08 ± 1.02	3.14 ± 3.89	2.31 ± 2.07
Group III	3.52 ± 4.92	2.04 ± 1.49	2.16 ± 1.37
Group IV	2.94 ± 4.32	2.47 ± 1.93	2.53 ± 1.85
Group V	9.63 ± 14.03	1.81 ± 1.48	2.06 ± 1.08
Group VI	2.73 ± 1.48	2.25 ± 1.31	1.2 ± 1.2

SD: Standard deviation, MPa: Megapascale

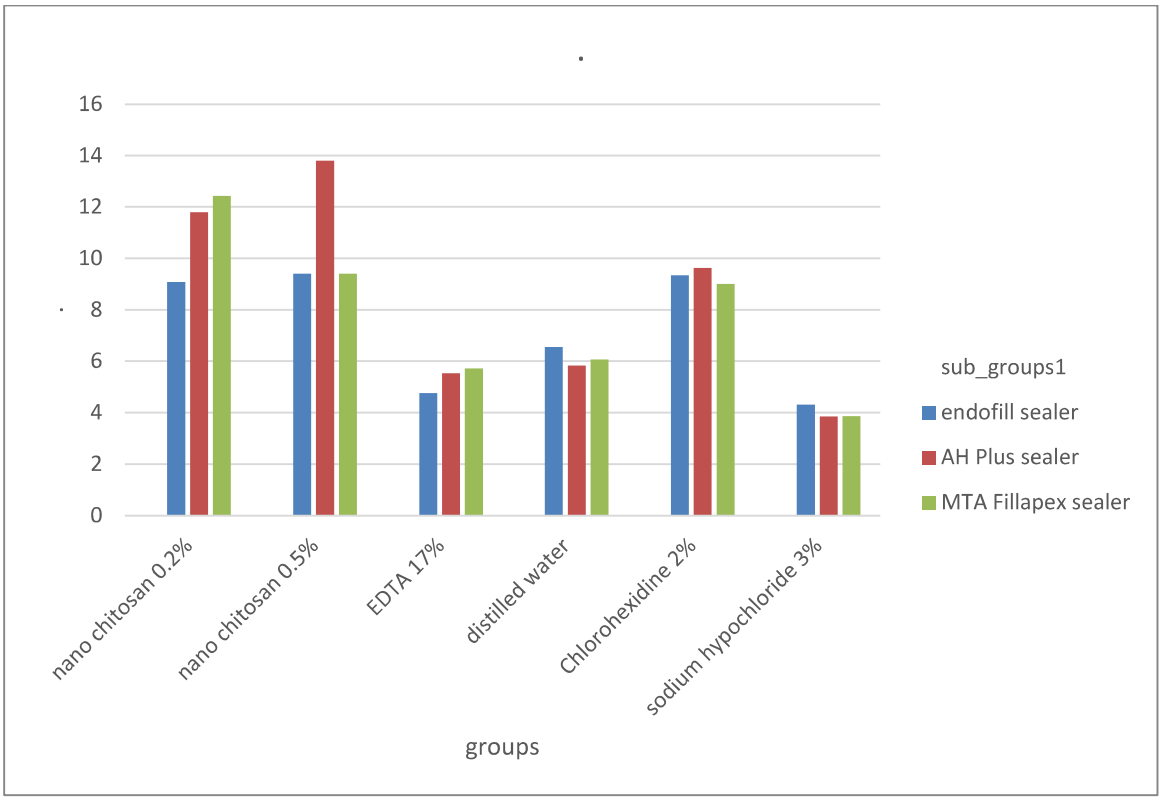

Figure (4.8): Histogram of the means POBS for each sealer A: AH Plus, B: MTA Fillapex, C: EndoFill with irrigation groups

4.2.2 Push Out Bond Strength of Each Irrigation Group at Three Levels of Root Canal Regardless of Root Canal Sealer

The highest mean values were showed at apical and lowest at coronal third. However, the difference between the mean values of all thirds were not significant. Regardless of root canal sealer used, the mean values were found to be statistically significantly higher within group 0.5% CNPs ($p<0.05$) when the groups were compared based only on the irrigation solutions. Other irrigation solution groups did not show any statistically significant differences table (4.8).

Table (4.8): Comparisons of POBS means for different root section at each experimental group alone

Groups	Root levels	Mean ± SD	p- value
G I	Apical	3.24 ± 2.98	0.67
	Middle	2.88 ± 2.59	
	Coronal	1.75± 2.89	
G II	Apical	4.67 ± 1.81	0.004 (**)
	Middle	2.19 ± 1.02	
	Coronal	1.95 ± 1.93	
G III	Apical	4.35 ± 2.08	0.071
	Middle	2.44 ± 1.76	
	Coronal	1.64 ± 1.25	
G IV	Apical	2.62 ± 1.86	0.49
	Middle	1.8 ± 1.77	
	Coronal	1.6 ± 1.41	
G V	Apical	3.59 ± 2.16	0.42
	Middle	2.62 ± 2.07	
	Coronal	1.53 ± 1.16	
G VI	Apical	3.16 ± 0.55	0.3
	Middle	2.43 ± 2.15	
	Coronal	1.72 ± 1.38	

SD: standard deviation

4.3 Failure Mode Analysis

The failure mode percentages are reported in table (4.9) for each experimental group at each section. The three forms of failure modes were visualized using stereomicroscope at 40x are seen in figure (4.9)

Table (4.9): Percentages of failure modes for the different groups at different root sections

Grs	Root levels	AH Plus			MTA Fillapex			EndoFill		
		Adhesive	Cohesive	Mixed	Adhesive	Cohesive	Mixed	Adhesive	Cohesive	Mixed
GI	Apical	33.3%		66.66%			100%	100%		
	Middle	66.66%		33.3%	100%			66.6%		33.3%
	Coronal	33.33%	33.33%	33.33%	100%			100%		
G II	Apical			100%			100%	100%		
	Middle	33.3%		66.6%	100%			33.33%		66.66%
	Coronal	33.33%		66.66%			100%	100%		
G III	Apical			100%			100%	33.3%		66.66%
	Middle	66.66%		33.33%			100%	100%		
	Coronal		66.6%	33.33%		66.66%	33.33%			100%
G IV	Apical			100%			100%	100%		
	Middle	100%				66.66%	33.33%			100%
	Coronal	33.33%		66.66%	33.3%	66.66%		100%		
G V	Apical			100%			100%	33.33%		66.6%
	Middle			100%	100%			100%		
	Coronal		100%		100%					100%
G VI	Apical	66.6%		33.3%	66.6%		33.33%	33.3%		66.6%
	Middle	66.6%	33.3%		100%			100%		
	Coronal	66.66%		33.3%	33.33%		66.6%	100%		

Figure (4.9): Stereomicroscope view mode of failures: (A) Adhesive failure, (B) Cohesive failure, (C) Mixed failure

CHAPTER FIVE
DISCUSSION

CHAPTER FIVE

DISCUSSION

The removal of pulpal tissue, bacteria, endotoxins, and proper shape of the restrictive dentin is a necessary part of the root canal preparation. It also creates space for a large volume of irrigants to flow through the canal to accomplish the goal of a successful cleaning and ready the canal to receive the filling material for an efficient apical seal therapy (Kareem and Kadir, 2022).

The development of rotary devices made of nickel-titanium (NiTi) in dentistry has brought about substantial changes to the root canal preparation process. Because they can prepare canals more quickly, cut more efficiently, reduce operator fatigue, maintain canal curvature, and reduce procedural errors like ledging and transporting, they have become more and more common over the past 20 years (Lall *et al*., 2021).

Pro-taper endodontic rotary files are designed with a progressive taper over the length of their cutting blades, allowing each instrument to prepare a specific area of the canal (i.e., each file engages a smaller zone of dentin). This file system has been selected in this study because this file may have a lesser tendency to pack debris into canal recesses, and thus may result in a higher push-out bond strength than the reciprocating files. Additionally, it is the most commonly used file system currently and gives satisfactory results (Chatterjee *et al*., 2021).

A gauge (30) double-side vented irrigation needles were used in this study. Due to their small bore size, these needles can inadvertently creep into the apical one-third of the canal, increase the irrigant's effective contact with the wall to remove SLs, and prevent the irrigants' vigorous passage through the apical foramen (Kuruvilla *et al*., 2015; Murugesan *et al*., 2022).

The study has used newly extracted human single roots which were about 16 mm long to standardize the samples in each group. They had a well-

developed apical constriction that prevented the intra-canal irrigation from flowing out the root canal and inserting the root canal sealer easily. To offer specimens that enabled the load to be properly aligned along the vertical axis of the root and yield pure forces during a testing method, teeth with straight canals were selected. To remove any variation between samples, the anatomical crowns were decoronated perpendicular to the root long axis to 12mm length using a continuous machine and diamond disc with plenty of water as coolant (Elbanna *et al*., 2019).

The apex of each tooth was covered with pink wax and placed in a polysiloxane elastomer impression material filled polyvinyl chloride (PVC) tube to replicate the anatomical scenario and provide a closed canal system that prevents extrusion of irrigation from the apical apex and permits recapitulation canal patency (Raouf and Saeed, 2020).

5.1 Smear Layer Removal:

In a previous study, Silva *et al*. (2012) and Darrag (2014) concluded that an irrigation of 0.2% chitosan for 3 min sufficiently cleared the smear layer, had better fallouts, and caused degraded dentine less than EDTA. This is contrasted with EDTA, which causes erosions and excessive removal of peritubular and intratubular dentin when used longer than 1 min. For easy and standardized comparison, the final irrigations were administrated in the same volumes for the same application time to all groups. The application time was selected based on a prior study, that suggested the application time of 1ml of chitosan and its nonparticles is 3 min (Mostafa *et al*., 2018).

In a study made by Ratih *et al*. (2020), it had concluded that 3 min application time produced better apical sealing ability and stronger bond strength than 1 min, independent final irrigation utilized. Thus, the application time of 3 min is chosen when using chitosan nanoparticles for the final irrigation solution. For greater sealing capability and binding

strength, the application of irrigations for a longer time is needed which is most efficient in clearing the apical third canal from smear.

According to a descriptive analysis, there was the highest smear layer present for GVI (distilled water), this is in agreement with previous research. This may be due to its solely intended to be as lubricant and whatsoever has no impact on microorganism or dentin smear layer. It was utilized as a control group for this reason (Suchithra *et al*. 2017; Mahdi and Baker, 2019).

The concentration of 5.25% of NaOCl is most effective among the three different concentrations 0.5%, 2.5%, and 5.25%. However, higher concentrations may cause ultrastructural damage to the dentin and could harm apical and periapical tissues. Therefore, a 3% concentration has been chosen for this study as it is less toxic and conveys less erosion, less collagen degradation, and is commercially available (Goel *et al*., 2022).

The GIV (3% NaOCl) in this study has higher smear layer removal than CNPs. This is due to acting as a nonspecific deproteinizing substance that can dissolve the organic phase of smear layer, this leads to thinning of smear layer which facilitates irrigation penetration (Yuan *et al*., 2023)

In a study by Nanda *et al*. (2023), when comparing the effect of irrigation of 3% NaOCl with saline which was used as a control on the smear layer removal showed 3%NaOCl was more efficient than saline. Sodium hypochlorite has oxidizing and hydrolyzing properties. Because of its strong proteolytic effect, it is a great aid during instrumentation. It is more effective for its ability to break down and degrade proteinaceous matter (Nanda *et al*. 2023). Sodium hypochlorite solutions consist of Hypochlorous acid, chemical components that behave like solvents upon contact with organic tissue. Also, it releases chlorine, which is inserted in a chemical process in contact with an amino group of protein, and the formation of chloramines that compromise the metabolism of microorganisms.

Kassaee *et al*. (2016) concluded that CNP appears to be a more effective permeable solution than 17% EDTA, NaOCl, and ordinary Chitosan (CH). Because of the small nano size of CNP, it has a low contact angle and higher penetration, under comparable conditions. Chitosan nanoparticles (CNPs) are a natural final irrigation solution, which have been used recently in endodontics because they have properties of bio-degradability, bio-compatibility to human cells, antibacterial, bio-adhesion, and lack of toxicity. CH and CNPs can be an alternative option instead of EDTA with these properties (Ilhan *et al*., 2024).

This study has employed chitosan in the form of nanoparticles because of its deeper absorption and penetration into dentinal tubules and intertubular dentin to increase the elimination of the smear layer. Because 0.5% chitosan nanoparticles were successful in eliminating smear layers in coronal, mid, and third apical root canals, this investigation used a chitosan percentage of 0.5% (Aydın *et al*., 2019).

CNP demonstrated better smear layer removal than CH, this implies that the decrease in particle sizes, stronger surface area, and higher chemical reactivity of CNP may have contributed to the improvement of its chemical and physical characteristics (Chandak *et al*., 2021).

Bajpe *et al*. (2023), concluded that a 0.2% CNP solution was better efficient than a 17% EDTA irrigant in eliminating a thin layer of debris at the apical third. This is consistent with the present study findings, 0.2% CNP (mean rank 20.17) more effective than 17% EDTA (mean rank 27.67) in smear layer removal at apical third and 0.2% CNP (MR 19.06, 19.26) has comparable effect as 17% EDTA (MR 20.39, 19.98) at coronal and middle thirds. Prior studies by Hassan and Negm (2018) and Hussein *et al*., (2022), found that a 0.5% CNP has a higher capability to eliminate the smear layer from the apical third than a 17% EDTA. This is in accordance with this study

showed that 0.5% CNPs were better at smear removal than 17% EDTA at apical third.

The main advantages of chitosan in endodontics are it's capacity to eliminate the smear layer and its chelating capabilities when utilized as an irrigation agent, which can lead to dentin erosion without affecting intertubular dentin. The mechanism of chelating action is unclear, but it is believed through absorption, ion exchange and chelation properties. There are two theories explaining the chelating process of chitosan, namely: (I) Chemical chain bridge model, two or more amino groups from one chain of chitosan will bind to the same metal ion; (II) the hook or free-arm model", which mentions that only one amino group of the material structure is involved in the binding, which is the metal ion attached to the amino group (Abidin *et al*., 2022). Chitosan polymer is made up of a chain of chitin dimers. This chitin dimer has two nitrogen atoms with free electron pairs that allow the metal to engage with the chelating chemical. In an acidic environment, amino groups exist in the protonated bipolymer, resulting in a full position charge (-NH3+). This structure enhances binding with other molecules, leading to the occurrence of adsorption (kamble *et al*., 2017; Ratih *et al*., 2020).

The apical portion of the root canal has a narrower diameter, making it more difficult to remove the smear layer. The dimension of CH nanoparticles can augment the elimination of SL by improving the penetration of irrigation fluid into DT. The chitosan polymer has hydrophilic properties. This facilitates its efficient adsorption by the root canal walls and promotes deeper delivery to the dentinal tubules through sustained contact with the root canal dentine. Furthermore, it possesses a substantial quantity of free hydroxyl and amino groups that facilitate ionic interactions between dentin calcium ions and the chelating agent (Aydin and Buldur., 2018; Hussein *et al*.,2022).

Oraim and Al-Zaka, (2024), they observed in their study that a 0.5% CNP demonstrated superior smear removal than a EDTA at the apical third

and as efficient as 17% EDTA at the coronal and middle third. This is in accordance with this investigation that shows a 0.5% CNP (MR 20.17) at apical thirdhaving a higher effect than 17% EDTA (MR 27.67) and at coronal and middle (MR 19.94, 19.16) as efficient as 17% EDTA (MR 20.39, 19.98). The lower capability of EDTA for eliminating the smear layer in the apical third may be attributed to EDTA having a property of chelating activity and reacting with calcium ions in dentine and forming soluble calcium chelates, and the apical third has less amount of water-soluble non-collagen proteins which EDTA ejects calcium from them, therefore less EDTA decalcification. EDTA decalcifies dentine to a depth of 20–30 microns in 5 min, thus higher concentration and prolonged contact time of solution which may increase cleaning but increase demineralizing of dentine. The effect of EDTA may be restricted by tubular sclerosis of dentin in the apical third (Karuppiah *et al*., 2021).

Statistical analysis of this study showed that 2% CHX had more smear layer surviving at all thirds than other experimental groups. This is consistent with the study of Dewi *et al*. (2020). Although CHX possesses a wide spectrum of antibacterial activity, substantively and lower toxicity than NaOCl but not able to resolve necrotic residues and remove the smear layer, so not utilized as a regular major irrigation (Thakur *et al*., 2020).

5.2 Push Out Bond Strength

The process of sealer adhesion to dentin and the core material (gutta-percha) is intricate, with distinct mechanisms for different sealer kinds and dentin conditions. The endodontic sealer's adhesive capability is gauged by its bond strength to dentin, often known as its dislodgement resistance. The success of endodontic therapy will be increased if there is a strong adhesive interaction between the sealer and the core material as well as between the sealer and the radicular dentine (Yaduka *et al*., 2021).

The main goal of root canal fillings is to stop the oral cavity and peri-radicular tissue from leaking into the root canal system. Gutta-percha is a biocompatible material used to fill radicular space, whereas a sealer is required to aggregate the filling material, preserve compact mass without voids, attach it to the canal wall, and give a single unit configuration. Bond strength testing is the most widely used method for assessing the effectiveness of the binding capability between radicular dentin and endodontic root-filling materials. Several irrigation schedules were used in the current study to assess their effect on the pushout bond strength of the AH Plus, MTA Fillapex and Endo Fill sealers with root canal obturation (Shivanand *et al*., 2020; Pawar *et al*., 2022).

The push-out test is commonly used to assess the bond strength between root canal sealer and root dentin because it provides a more accurate assessment of bond strength (Madhuri *et al*., 2016).

It was necessary to standardize the push-out test in order to look into specific issues related to the sealer–dentin interface. The thickness of the root sections, the plunger's size in relation to the root canal's diameters, and the specimen's orientation can all affect the push-out test's results and validity. The plunger tip's dimensions ought to be less than those of the filling material. Additionally, the plunger tip's position needs to be closer to the sealer's diameter. The fracture profile and stress distribution are directly impacted by the load applicator tip's diameter. A load with a diameter significantly bigger than the root canal's tends to induce the sample to flex, increasing dentin stress and causing failure before it happens at the adhesive interface. On the other hand, because they create more strains in the filling material, small-diameter load applicator tips mostly result in cohesive failure (Neelakantan *et al*., 2018; Dem *et al*., 2019; Rodrigues *et al*., 2021; El Sayed and Alderei, 2023).

Three plunger sizes (0.3, 0.5, and 0.7mm) corresponding to (apical, middle, and coronal) sections respectively were employed for each root third

in the current investigation in order to fully cover the core material in each third of the sample and minimize the impact of that variable. To prevent any constriction interference from the root canal taper, the force was supplied to the obturating material apically to the coronal direction, and the plunger was centralized to prevent contact with the dentin. During slicing, thin slices (approximately 1-2 mm) run the danger of the sealer coming off. To prevent premature debonding, slices of 2 mm thickness were used in this investigation (Chole *et al.,* 2019; Elmuttalibi and Mahdi, 2019).

The removal of the smear layer increased the bond strength of AH Plus to root dentin but negatively affected on calcium silicate base sealer as it facilitates exposure of the collagen network and improves AH Plus sealer penetration into the dentinal tubules but calcium silicate-based bioceramic sealer is a hydrophilic material that uses the moisture in the smear layer to form a hydroxyapatite-like precipitate during setting and form a chemical bond with dentin. The moisture present in dentinal tubules after dryness is not enough to set the sealer and reduce the bond strength (Mohamed *et al*., 2021)

MTA Fillapex had higher bond strength when the smear layer was maintained than when it was not. This relates that the smear layer's moisture has a beneficial effect on MTA Fillapex's adherence to the canal wall because MTA-based materials can release calcium and hydroxyl ions that form hydroxyapatites through a process known as biomineralization when they come into contact with fluids containing phosphate. By depositing these apatites in the collagen fibrils, they encourage the formation of an interfacial layer known as a mineral infiltration zone and tag-like structures between MTA and radicular dentin, which can result in increased push-out bond strength values (Türker *et al.,* 2018).

As shown above chitosan in the endodontic is its ability to remove the SL and has chelating properties when used as an irrigation agent and can cause dentine erosion but not intertubular dentine. This study employed

chitosan in the form of nanoparticles because of its deeper absorption and penetration into dentinal tubules and intertubular dentin in order to increase the elimination of the SL and increase infiltration of sealer (Aydın *et al*., 2019).

Chelating drugs alter the ratio of calcium to phosphate in tooth structure in addition to removing the smear layer that is rich in calcium and phosphate may adversely affect the adherence of calcium silicate-based sealers, which primarily rely on the calcium ions found in dentine for the biomineralization process. CNPs had less adverse effects on the bond strength and adherence of bioceramic sealer compared to EDTA because chitosan results in less erosion as well as less alteration in surface structure and ca\p ratio (Carvalho *et al.,* 2016; Mathew *et al*., 2017; Mohamed *et al*., 2021).

The current study stated that 0.2% and 0.5% CNPs have a higher effect on push-out bond strength than 17% EDTA this is in disagreement with the prior studies which have concluded that 0.2% chitosan has produced the same bond strength as 17% EDTA (Chaves *et al*., 2019).

CNPs improve the bond strength of AH Plus sealer. The effect of CNP in clearing the smear layer may be due to the chelating capability and hydrophilicity nature of CNP that made it in intimate contact with radicular dentin, quickly adsorb into canal wall, and insert deeply in dentinal tubules. Also, the cationic property of CNP facilitates interaction between the calcium ions present in the dentinal wall and chelating chemicals. Protonation of the chitosan's amino group causes other molecules to withdraw for adsorption into the root canal dentin, allowing the substance to enter the dentinal tubules more deeply. Conversely, the hydrophilic character of chitosan can enhance the wetting ability of sealer material on the root canal wall, which has an uneven surface because of the dentinal tubules opening following instrumentation and irrigation. This is because AH Plus sealer is hydrophobic. Additionally, chitosan particles that are nanosized can

accelerate the flow of irrigation solution into the dentinal tubules, causing the smear layer to be removed. This allows the obturation material and root canal wall to bind with sufficient strength (Ratih *et al*., 2020; Geogi *et al*., 2023).

According to the current investigation, an MTA Fillapex sealer's resistance to dislocation is enhanced by final watering with CNPs. This is consistent with a prior study that found that a 0.2% chitosan final irrigation enhances an MTA resin hybrid root canal sealer's resilience to dislocation. To improve the stability of MTA bonds, chitosan has been utilized for extra fibrillar dentin demineralization. This tactic has been suggested to stop water penetrating hybrid layers and lessen endogenous collagen breakdown caused by matrix metalloproteinases (MMP). Recent data indicates that the chelating ability of chitosan, not the solvent's function, is responsible for demineralizing extra fibrillar dentin, despite a body of research suggesting that the acetic acid solvent is the reason for chitosan's effectiveness in calcium ion dissolution (Gu *et al*., 2018; Ozlek *et al*., 2020).

Kaki and Genç Şen, (2018). Concluded that a 17% EDTA improved the bond strength of AH Plus and chitosan did not improve the push-out bond strength of it. Our research revealed that group III's mean push-out bond strength (17% EDTA) has been lower than that of groups I and II (0.2% and 0.5% CNP). This could be explained by EDTA's lesser viscosity compared to chitosan and its limited ability to penetrate sealers. Therefore, 17% EDTA may enter dentinal tubules, eliminating smear clogs and allowing AH plus sealer to enter. This result is consistent with a prior study that found that AH plus sealer penetration was minimal following a final rinse with a 17% EDTA. EDTA irrigation results in thick demineralized collagen matrices that may collapse when dried with paper, preventing full resin infiltration (Hashem, *et al*., 2019; Medhat *et al*., 2022).

When the EDTA has been employed as a last irritant before applying the AH Plus sealer, noticeably poor push-out bond strength has been noted.

The lack of a surfactant effect and limited demineralizing capacity of the 17% EDTA serves as an evidence for this. Consequently, a very thin layer of demineralized collagen fibrils has been developed on the surface of dentine. The AH Plus root canal sealer's poor wettability on the 17% EDTA-irrigated dentine is caused by this layer. When resin sealers are irrigated with 17% EDTA, their contact angle increases in comparison to other studied irritating materials (Rifaat *et al*., 2023)

In this study, EDTA reduces the bond strength of MTA Fillapex. This result is in accordance with the prior study which stated that EDTA reduces the bond strength of MTA sealer. This may be attributed to the fact that residual EDTA in the root canal dentin continues to chelate calcium ions released from MTA during hydration, interfering with the precipitation of the hydrated products. EDTA has a negative effect on the microhardness and hydration of MTA. A 17% of EDTA reduces the bond between the bioceramic sealer and the dentine wall of the root canal. This could be due to the fact that EDTA affects the apatite that forms during the sealer's setting reaction (Naeem and Al-Zaka, 2021).

Veeramachaneni *et al*. (2022), concluded that 17% EDTA disrupts the connection between bioceramic and radicular dentine. This might be because calcium silicate's hydration can be disrupted by EDTA's chelation of calcium. Reduced contact between the sealer and the root canal wall could arise from the depletion of calcium at the sealer–dentin interface or from the breakdown of the calcium silicate component in the sealer, which could prevent the creation of the mineral infiltration zone.

Group VI (distilled water) has displayed lower mean bond strength values, and all sealers have displayed lower bond strength values when irrigated with distilled water due to the sealers' inability to reach the dentinal tubules. Since distilled water contains a large amount of water, it did not eliminate the smear layer, which has an unknown thickness and volume (Bayram *et al*., 2017).

Group IV (3% NaOCl) results in the lower push-out bond strength, this may be the consequence of the lower molecular weight NaOCl's ability to pierce the apatite-matrix-covered collagen and remove organic materials from the mineralized dentine. The hybrid layer on the AH Plus sealer has been harmed, and the density and adaptability of the sealer material into the dentinal tubules have decreased due to the damaged dentin structure (Xu *et al*., 2022).

The dislocation resistance of AH Plus to root canal dentin is significantly decreased in this research by using NaOCl as a final irrigant, which is consistent with study of Elmessiry *et al*. During instrumentation, NaOCl is applied to the canal to remove the organic. Collagen or other dentin matrix components may be oxidized by the production of oxygen bubbles at the resin-dentin interface when NaOCl breaks down to sodium chloride and oxygen. This results in a layer of oxidized dentin that strongly inhibits the interfacial polymerization of resin-bonding materials and obstructs resin infiltration into tubules and intertubular dentin (Elmessiry *et al*., 2019). The adherence of AH Plus sealer, which depends on the creation of covalent connections between its open epoxide ring and any exposed amino groups in collagen, is also negatively impacted by NaOCl, a deproteinizing chemical that can degrade dentin by dissolving collagen. Additionally, the hydrophilic surface created by NaOCl deproteinization counteracts the sealer's hydrophobicity (Khanvilkar *et al*., 2023).

The foundation of MTA-based sealers is calcium silicate and resin salicylate. It is anticipated that resin sealers and MTA-based sealers will have some similarities in their bond strength to dentin given their chemical makeup. NaOCl weakens the MTA Fillapex bond (Nafiz *et al*., 2018). It is believed that NaOCl causes the dentine matrix to oxidize, producing protein-derived radicals that would hinder the spread of vinyl free radicals in resin, leading to partial polymerization and a decrease in calcium and phosphorus

levels which have a negative effect on the adhesion of MTA Fillapex, which depends on calcium and phosphorus for the biomineralization (Attash and AL-Ashou, 2022; Grazioli *et al*., 2024).

In the present study, a 2% CHX and 0.2% CNPs have shown comparable results in the coronal and middle sections of AH Plus and MTA Fillapex. The higher bond strength obtained with CHX group for both sealers be attributed to various factors. CHX gets adsorbed into the root dentin and released as long as 48 to 72 hours after irrigation; this could have favored resin infiltration into dentinal tubules and have a positive effect on the bond strength (Costa *et al*., 2021). Additionally, CHX makes endodontic sealers more wettable on dentin. This is because CHX contains surface surfactant, which raises surface energy, encourages a higher wetting ability to dentin and reaction of the polycarboxylic group enhancing the cationic charge. This property is required for the adhesion of bioceramic sealers due to their hydrophilic nature. Furthermore, by inhibiting matrix metalloproteinase MMPs and preserving the collagen matrix, CHX creates a more stable and favorable environment for resin monomers to penetrate the dentinal tubules (Celikten *et al*., 2022). MMPs are enzymes that can degrade the collagen fibers within the dentin matrix, weakening the bond between the resin and dentin. CHX (Coelho *et al*., 2020).

When CHX has been applied as the last irrigant, AH Plus has been the best performer, displaying the highest push-out bond strength values in the coronal one-third of the root. While when an MTA-based sealer is used it has shown a significantly higher bond strength in the middle one-third as opposed to be coronal one-third. The main cause of high push-out bond strength may be because CHX strengthens the resin-based sealer's bond, and the coronal third has a lot of dentinal tubules. More resin penetration and resin tag production will occur in the presence of many dentinal tubules. In exchange, this results in a stronger sealer bond. This is in accordance with a study of Maan, *et al*., (Maan, *et al*., 2022).

In this study, AH Plus has higher POBS than MTA Fillapex. This is in accordance with previous studies. AH-Plus demonstrated the strongest bond across all cross-sections in all groups of this study, while MTA-Fillapex has came second for bond strength in the middle and apical root thirds. In the apical third, MTA-Fillapex demonstrated a bond strength that was comparable to that of AH-Plus in all groups which is consistent with the study of Abdollahi *et al* , this may be explained by that Low polymerization shrinkage, long-term dimensional stability, the covalent linkage between epoxide (open circle) and the exposed amino groups in collagen and inherent volumetric expansion have all been credited with the greater bond strength values linked to epoxy resin sealers (Abdollahi *et al*., 2024). On the other hand, MTA Fillapex may have a weaker bond because of MTA-Fillapex release calcium and hydroxyl after its setting result in formation of apetite. This apetite precipitate among collagen fibrils to form tag-like structure so maker adhesion of this tag-like structure which assumedto compromise the root canal seal (Kurup *et al*., 2021). The bonding behaviors of MTA-based sealers can be affected by their chemical compositions. Resistance of MTA against dislodgement is promoted by its biomineralization capacity. However, due to the presence of resin components in MTA Fillapex sealer structure and decreased adhesion of apatite tag-like structures, a reduced bond strength to dentin willbe achieved. Their sealing and adhesion properties might be influenced by their solubility as well (Yavari *et al*., 2017)

The EndoFill group had the lowest mean bond strength in the coronal, middle, and apical thirds, according to the study's findings. One explanation could be that Grossman sealers, or sealers containing ZOE base, adhere to dentin by electrostatic bonding rather than by penetrating the dentinal tubules. Both the zinc oxide component of gutta-percha and the mineral composition of dentin may react with the zinc ion in zinc oxide. Furthermore, gutta-percha may become softer as a result of eugenol. As a result, a crisscross design is produced, which improves the materials' ability to stick

together. Gutta-percha contains zinc oxide, which chelates the eugenol component of zinc oxide eugenol sealers (Abdollahi *et al*., 2024). Inappropate polymerization of these sealers lead to stress on the root canal walls can lead to marginal gaps, microleakage, and a few clinical failures. A strong bond with the root canal dentin cannot be created if one side of the root canal separates due to forces of polymerization shrinkage that may be greater than its bond strength to the root dentin (Gündoğar *et al*., 2016; Aakriti *et al*., 2019).

The root canal filler's resistance to displacement in the coronal to apical direction has increased. Regardless of the irrigation protocol and sealers used, the results of the current investigation's analysis of POBS means across root levels revealed statistically significant variances The highest mean was observed at the apical level and the lowest at the coronal level. This should be put into perspective with the anatomy of the root canal, the film thickness of the sealer and the selected root canal filling technique. It is well known that the root canal cross-section in the apical third is relatively round compared with the oval shape of the canal in the coronal third. Likewise, when applying a single-cone technique (with no intra-canal gutta-percha compaction), the film thickness of the sealer is necessarily greater in the coronal and middle third of the root canal than in the apical third where the master cone is well fitted. These considerations may explain the greater POBS values in the apical third regardless of the sealer type and irrigation protocol (Verma *et al*., 2018; Sfeir *et al*., 2023).

CHAPTER SIX
CONCLUSIONS AND SUGGESTIONS

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6.1 Conclusions

Under the limitations of this study, we concluded:

1. 0.2% and 0.5% CNPs irrigations are the more effective in eliminating smear layer than other tested irrigation solutions at apical level of root canal.
2. 0.2% and 0.5% CNPs have comparable effect to 17% EDTA in eliminating smear layer at middle and coronal levels of canal.
3. 3% NaOCl irrigant is the most effective in eliminating smear layer among the tested irrigations at coronal and middle levels.
4. 2% CHX and distilled water have the least effect on smear layer removal at all levels.
5. CNPs irrigations have a positive outcome on push-out bond strength of all tested sealers.
6. 0.2% and 0.5% CNPs have a higher effect on push-out bond strength than 17% EDTA.
7. 17% EDTA have a poor effect on push-out bond strength of AH Plus and MTA Fillapex sealers
8. 3% NaOCl irrigation didn't improve the push-out bond strength of all tested sealers.
9. 2% CHX improves the push-out bond strength of AH Plus and MTA Fillapex sealers.
10. Distilled water has the least effect on the push-out bond strength of all tested sealers.
11. AH Plus sealer have a higher push-out bond strength than MTA Fillapex sealer while EndoFill sealer have the least bond strength at all levels of all irrigation solutions.

6.2 Suggestions:

1. Evaluation of the effect of CNPs on dentine degradation, microhardness of dentine, and roughness.
2. Evaluation of the effect of CNPs incorporated with other irrigation solutions on smear layer removal, and cytotoxicity.
3. Assessment of the effect of different application times of CNPs on smear layer removal and push-out bond strength of sealers.
4. Assessment of the effect of different concentrations of CNPs on smear layer removal and push-out bond strength of sealers.
5. Evaluation of the effect of CNPs incorporated with other irrigation solutions on push-out bond strength of sealers.
6. Examine the impact of varying application times of chitosan nanoparticles combined with other irrigation solutions on smear layer elimination and the push-out bond strength of sealers.
7. Evaluation of the effect of CNPs on Enterococcus faecalis microorganism.

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الخلاصة

الأهداف: تهدف هذه الدراسة المختبرية إلى تقييم تأثير محلول جسيمات الشيتوزان النانوي بتركيزين (٠.٢٪ و ٠.٥٪) على إزالة طبقة اللطاخة وقوة الترابط وأنماط الفشل في مواد مانعات التسرب (AH Plus, MTA Fillapex, EndoFill) على العاج الجذري، ومقارنتها مع محاليل أخرى.

المواد والطرق: تم الحصول على إجمالي ١٠٨ سن ضاحك سفلي بشري ذات جذر مستقيم واحد. تم قطع تاج جميع الأسنان لترك طول جذر ١٢ملم. تم الوصول إلى كل جذر وتحديد طول السن. تم تحضير القنوات باستخدام مبارد دوارة (Pro-Taper) الى حجم F3 خلال التحضير وبين كل مبرد تم غسل القناة بـ ٢ مل من 2.5% NaOCl متبوعًا بـ ٥ مل من الماء المقطر لإزالة تأثيرها. بناءً على الغسل النهائي تم تقسيم العينات إلى ست مجموعات (n=18) وتم غسل القنوات بحوالي ٣ مل لمدة ٣ دقائق من كل غسول نهائي. المجموعة الأولى: ٠.٢٪ محلول جسيمات الشيتوزان النانوي، المجموعة الثانية: ٠.٥٪ محلول جسيمات الشيتوزان النانوي، المجموعة الثالثة: ١٧٪ حمض الإيثيلين ثنائي امين رباعي الأسيتيك، المجموعة الرابعة: ٣٪ هيبوكلوريت الصوديوم، المجموعة الخامسة: ٢٪ كلورهيكسيدين، والمجموعة السادسة: الماء المقطر كمجموعة سيطرة.

تم أخذ تسع عينات من كل مجموعة لاختبار إزالة طبقة اللطاخة، وتم تقسيم كل عينة طوليًا وملاحظتها تحت المجهر الإلكتروني الماسح (SEM) عند تكبير 2000X . أخيرًا، تم تحليل البيانات المجمعة إحصائيًا. تم استخدام تسع عينات أخرى من كل مجموعة لاختبار قوة الارتباط بالدفع وتم توزيعها الى ٣ مجموعات (n=3) على اساس استخدام مادة مانعة التسرب اللبية. المجموعة أ: AH Plus، المجموعة ب: MTA Fillapex، المجموعة ج: EndoFill. تم استخدام جميع المواد المانعة للتسرب وفقًا لتعليمات الشركة المصنعة. تم سد قناة كل عينة باستخدام تقنية المخروط الفردي وتم تحضينها لمدة ٧ أيام عند ٣٧ درجة مئوية ورطوبة ١٠٠٪ في الحاضنة. تم قطع ثلاث شرائح بسمك ٢ ملم من كل عينة من المستويات التاجية والوسطى والقمعية. تم استخدام آلة اختبار عالمية لتحليل قوة الارتباط بالدفع (POBS) بسرعة رأس متقاطعة ١ ملم/دقيقة، وتم استخدام المجهر المجسم بتكبير 20X لفحص نمط الفشل. ثم تم جمع البيانات وتحليلها إحصائيًا باستخدام اختبار Kruskal-Wallis واختبار Mann-Whitney واختبار Post-hoc لاختبار إزالة طبقة اللطاخة واختبار Kruskal-Wallis Test لاختبار قوة الارتباط بالدفع.

النتائج:

تم العثور على فروق ذات دلالة إحصائية بين المجموعات في جميع الأثلاث الثلاثة ($p < 0.01$) في اختبار Kruskal-Wallis عند المقارنة بين مجموعات جسيمات الشيتوزان النانوي ٠.٢٪ و ٠.٥٪ مع مجموعات الاختبار الأخرى، ولم يكن هناك فرق ذو دلالة إحصائية بين جسيمات الشيتوزان النانوي ٠.٢٪ و ٠.٥٪ ($p > 0.05$) في اختبار Mann-Whitney.

كانت جسيمات الشيتوزان النانوي فعالة للغاية كمحاليل مخلبية في الثلث القمعي، ولكنها كانت بنفس الفعالية في الأثلاث التاجية والوسطى مثل 17% EDTA. كانت كفاءة 3% NaOCl في الثلثين التاجي والوسط أعلى من جسيمات الشيتوزان النانوي. كان 2% CHX والماء المقطر الأقل كفاءة في إزالة طبقة اللطاخة في جميع الاثلاث.

لم يظهر اختبار Kruskal-Wallis أي فرق إحصائي بين المحاليل على تأثير قوة الارتباط بالدفع لمواد مانعة التسرب الثلاثة. كانت أعلى قوة ارتباط بالدفع في الثلث القمعي لجسيمات الشيتوزان النانوي، تليها 17% EDTA ثم 3% NaOCl . 2% CHX زاد من قوة الارتباط بالدفع لمادة AH Plus في الثلث التاجي ولمادة MTA Fillapex في الثلث الأوسط. كانت أدنى قوة ارتباط بالدفع لـ 3% NaOCl والماء المقطر.

الاستنتاجات:

تظهر النتائج أن محلول جسيمات الشيتوزان النانوي الجديد كمحلول غسل نهائي فعال للغاية في إزالة طبقة اللطاخة، خاصة في الثلث القمعي، مقارنة بـ 17% EDTA. كما يعمل محلول جسيمات الشيتوزان النانوي على تحسين قوة الارتباط لمواد مانعة التسرب AH Plus و MTA Fillapex و EndoFill بشكل أفضل من 17% EDTA.

يعتبر 2% CHX فعالًا مثل محلول الشيتوزان النانوي في تحسين قوة الارتباط لمواد مانعة التسرب AH Plus و MTA Fillapex و EndoFill، بينما يؤثر 3% NaOCl سلبًا على قوة الارتباط بالدفع لمواد مانعة التسربالثلاثة . جميع محاليل الغسل تؤدي إلى تقليل قوة الارتباط لمادة EndoFill ماعدا 0.2% و 0.5% محلول جسيمات الشيتوزان النانوي.

جامعة الموصل
كلية طب الاسنان

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رسالة تقدمت بها

فاطمة شامل محمد صالح حسن

الى

مجلس كلية طب الاسنان في جامعة الموصل

وهي جزء من متطلبات نيل شهادة الماجستير

في

علاج الأسنان التحفظي

بإشراف

الأستاذ المساعد

فنار تركي عبد الحميد الجدوع

١٤٤٦هـ ٢٠٢٥م