

Synthesis, Properties, and Dental Applications of Tricalcium Silicate: A Comprehensive Review

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ABSTRACT

Background: Tricalcium silicate (Ca_3SiO_5) is a key component in bioceramic materials used in endodontics and restorative dentistry due to its bioactivity, biocompatibility, and excellent sealing ability. It plays a crucial role in pulp capping, root-end filling, and dentin regeneration, making it a preferred material in modern dental applications. **Objective:** This review provides a comprehensive analysis of tricalcium silicate, covering its synthesis, physicochemical properties, and clinical applications in dentistry. It also highlights its role in tissue healing and its advantages over traditional dental materials. **Methods:** A literature review was conducted using scientific databases, focusing on studies related to the synthesis, structural properties, bioactivity, and clinical performance of tricalcium silicate-based materials. The review emphasizes its use in dental procedures such as pulp therapy, root repair, and mineralized tissue formation. **Results:** Studies confirm that tricalcium silicate-based materials exhibit superior sealing ability, high pH, antimicrobial properties, and excellent biocompatibility compared to conventional materials. Additionally, its interaction with tissue fluids promotes hydroxyapatite formation, enhancing dentin remineralization. Recent advancements in nano-engineered tricalcium silicate formulations have further improved its mechanical strength, setting time, and bioactivity, making it an ideal material for vital pulp therapy and endodontic repair. **Conclusion:** Tricalcium silicate remains a gold standard in bioceramic-based dental materials due to its enhanced biological and physicochemical properties. Ongoing research in material modifications and clinical applications will further refine its role in dentistry and regenerative medicine.

Keywords: Tricalcium Silicate, Bioactivity, Biocompatibility, Ca Ions Release, Healthy Environment to Enamel Substance

INTRODUCTION

Portland cement is typically composed of a synthetic rock called clinker, which minimally consisted of four main phases: the first two phases are alite and belite, both of which belong to the calcium silicate compound. While the other two phases are the aluminate phase and ferrite phase, alite phase forms about 50-75% of the clinker rock, which is the impure form of the tricalcium silicate Ca_3SiO_5 (TSC) [1, 2]. Industrial production of Portland cement occurred by firing the mixture composed of clay and limestone in a cement kiln at approximately 1450° C, this will produce the artificial rock-like material termed clinker which acts as the main component of the Portland cement [3]. In pure TCS the molecular orientation and crystallo-graphic configuration changes according to temperature, this phenomena explains why there are seven polymorphs belong to TCS. According to the above the TCS in room temperature till 620°C present as triclinic T1, after this temperature to 920°C the TCS will become triclinic T2, with more elevation in temperature below the 980°C the TCS will become triclinic T3. At temperature 980°C the TCS will converted to monoclinic M1, further increase in temperature to 990°C, the TCS will become monoclinic M2 phase, at temperature 1060°C the TCS will converted into monoclinic M3, with more ten degree increase in temperature the TCS with converted from M3 to rhombohedra phase R [4, 5]. All these phase can be stabilized in room temperature by adding trace element like Zink, ferrous, aluminum, and magnesium [2, 6] while the T1 the only pure form that present stable in the room temperature [7]. When the TCS is mixed with water, it will react, set and harden due to the development of the hydrated calcium silicate gel (CSH), with calcium hydroxide nucleates [8]. The manner endodontic therapy is delivered has changed significantly during the past 200 years. The conventional process has undergone several revisions, particularly as patient demand to save their teeth has

increased and as material science and novel equipment have advanced. Endodontic use of bioceramic materials namely TCS might be viewed as a remarkable development that has altered the outlook of numerous instances that were previously thought to be hopeless [9]. Best examples of the TCS derivative are the Calcium silicate cement (CSCs), which exhibit self-setting activity in a moist environment with water, blood, or other body fluids and are important dental cement for clinical use have received a lot of attention in recent decades as endodontic materials [10]. CSCs are materials with hydraulicity that consist of up of roughly 15%–30% Ca_2SiO_4 (C2S), 5%–10% $\text{Ca}_3\text{Al}_2\text{O}_6$, and 3-6% gypsum, as well as 50%–75% Ca_3SiO_5 (C3S) or (TCS), according to Meschi et al, [10] inorganic cement based on silicate is utilized as a material with high potential for root canal therapy [11] because they have suitable physical qualities, the capacity to induce cell proliferation and differentiation, as well as excellent bioactivity [10, 11]. Calcium silicate-based biomaterials can induce the mineralization of hydroxyapatite (HAp) when soaked into simulated body fluid. Silicon ion derived from the cement has a significant effect on promoting osteoplastic cell growth, differentiation, and osteogenic gene expression [12]. TCS has been utilized for bone tissue repair by itself or with additional compositions [11], in late 1990 Dr. Mahmoud Torabinejad and colleagues at Loma Linda University invented mineral trioxide aggregate MTA, a rare biocompatible polymer with unique medicinal uses. TCS forms the main constituent of the MTA which is a versatile material that can be utilized in both surgical and non-surgical procedures, such as direct pulp capping [12], temporary filling material, perforation repairs in roots or furcations [13], apexification, and root end fills [14]. Despite this miracle cement's high clinical performance, there were always a few problems that made it impossible for physicians to employ it in many situations. The two

main ones are difficult manipulation and a very long setting time [14, 15]. In 2009, the field of dentistry has seen the introduction of Biodentin novel bioactive calcium silicate-based cement as a "dentin substitute." To crystallize and interlock with dentin and give mechanical qualities, this new biologically active substance helps it pass through dentinal tubules that have been opened. As a result of adopting MTA-based cement technology in the formulation of Biodentin, the company claims improvements to a number of its features, including handling and physical qualities, as well as a wide range of other uses, including endodontic repair and pulp capping in restorative dentistry [16, 17]. TheraCal LC, a new light-cure tricalcium silicate resin-modified material, was introduced to the market in 2011 for use in both direct and indirect pulp capping. It has many benefits, including the ability to adhere to composites and thereby reduce micro leakage, a notable calcium release that aids in repairing dentin bridge formation, and user-friendliness [18]. Another commercial product based on TCS is the BioRoot RCS (Septodont) which enter the market in 2015 [15]. Several cement products launched to the market later based on TCS for example Endosequence BC Root Repair Material (Brasseler, Savannah, USA), which is user friendly due to pre-mixed modality and inject ability [19]. It is worth saying that all the TCS derivatives cement behaves differently whether they use with water or they mixed with organic resin-like Bis-GMA-based resin. TCS derivatives cement undergoes a hydration reaction when mixes with water, but this reaction will never occur when TCS derivatives used with resins, even so in both case the TCS capable to release calcium ions in biological solutions and encouraging deposition of calcium phosphate [20]. According to the above and as shown in table one the TCS material was used as main material used in dental practice namely, root apexification, endodontic sealer, pulp capping, pulpotomy, lining material, surgical and non-surgical procedures, such as, perforation repairs in roots or furcations and root end fills. This review of the literature aims to illustrate briefly the preparation, properties, behavior, and use of TCS in dentistry.

Preparation of the TCS

The nanoparticles TCS can be produced by several methods, but the most popular methods, are the solid state reaction (SS method), sol-gel method (SG methods), and Pechini method. In the SG method the TCS shows shorter setting time with more alkaline environment and better hardness, than the SS method. TCS behavior is depended on the method of preparation because crystalline phases of TCS depend on method of preparation and calcined temperature. This behavioral difference was confirmed by both X-ray diffraction XRD and fourier transform infrared spectroscopy FTIR [15, 21].

The Solid-State Method (S1)

In this method three parts of CaO were mixed with one part of the quartz SiO₂ the mixer was fire at 1000°C in presence of 0.5% boric acid as a catalyst. The item was pulverized, remolded using carbon tetrachloride, and burned for two hours at 1450° C. The firing cycle was repeated until the reaction was finished. The presence of free lime was examined in the finished product [21].

Sol-gel Method (S2)

TCS was produced by sol-gel technique, by a reaction between Tetraethyl orthosilicate (TEOS), also known as Si(OC₂H₅)₄, and calcium nitrate tetrahydrate (Ca(NO₃)₂·4H₂O) [22, 23]. A solution of

0.5 mol TEOS in 200 ml water was gradually supplemented with 1.5 mol of Ca(NO₃)₂·4H₂O while being continuously stirred. Nitric acid should be used to bring the pH of the solution to 4.5. The mixture was heated to between 50 and 55 °C for two hours, and the solution temperature was kept at 75 °C for twenty-four hours. The produced gel was heated up at 120 °C overnight and then calcined for nine hours at 1500 °C with a firing rate of 10 °C/min [24].

Pechini Method

The calcium source was Calcium nitrate tetrahydrate, (Ca(NO₃)₂·4H₂O) and the silicon source was colloidal silicon oxide SiO₂ (40wt% suspension in water) in Pechini method. Two raw materials with a Ca to Si molar ratio of 3:1 were dissolved into pure water. Citric acid was dissolved in water first, then it will add to the above cationic solution at a 1:1 molar ratio, the combined solution was agitated until it turned transparent. Ethylene glycol (C₂H₆O) was then added to the mixture at a molar ratio of 2:1 for Ethylene glycol to Citric acid. To evaporate the excess water and create a frothy gel, the finished mixture was heated to 80 to 100 °C while being stirred. The drying cabinet (150 °C) was used to dry this product overnight. The xerogel was crushed into powder and then heated in a furnace at 1200, 1300, and 1400 °C for 3 hours while increasing the temperature by 10 °C/min. The finished substance was milled into a fine powder [15].

X-Ray Diffraction XRD

This test regards as the most reliable and directs method to quantify and /or detect the TCS cement phases. Additionally, it can detect any additive and/or impurities material incorporated with TCS. As we mention before the TCS present in seven phases depending on the atomic and/or molecular (Ca, Ca-Si) arrangement and position within the crystal, method of preparation and temperature. exactly explains why literatures are showing variable XRD plots, and there is no uniform plot for the TCS [1-3]. TCS can't dissolve in water but react with it and undergo hydration and setting later on by the development of the calcium silicate hydration gel, C-S-H and lime as calcium hydroxide; the last also can be detected by the XRD [15]. TCS produced by the Solid state method (S1) shows slightly different plots to that TCS produced by sole gel methods (S2) in XRD figure 1, while both of them have different XRD plots depending on hydration or anhydration state. The Hydration state showed a reduction of the main peaks of TCS (2θ = 28. -29.5) with progression in time till 28 days, indicating that the unreacted TCS will undergo hydration reaction with time, and the curing of the hydrated TCS last for 28 days. The XRD plot of the S2 shows more declination TCS peaks upon hydration reaction than that of S1. The XRD main peak of the Ca(OH)₂ at 2θ = 18-19 is more well-demarcated in hydrated S2 than that of S1 [7, 21, 25].

The XRD peaks SI anhydrous state were mainly ranged at 2θ = 28-35 and 50-53, [11] the XRD patterns of anhydrous S2 are more temperature sensitive and depend, so the TCS peaks can be evident when the calcination temperature is between 1300-1450 °C, while the calcination temperature below 1400°C, will make the peak of Cao more clear evident at the 2θ = 54 [26]. TCS was prepared by Pechini Method (PTCS). Again, the temperature plays a role in XRD plot behaviors so when calcinated temperature reaches 1200 °C, we can see a plot of the TCS and CaO. Temperature elevation to 1300°C, the powder XRD plot will show mainly TCS with a small quantity of CaO. Further temperature elevation up to 1400°C, the XRD plot of this powder will

become mainly belong to TCS and a trace of CaO, which is much less

than in temp 1300 °C [15].

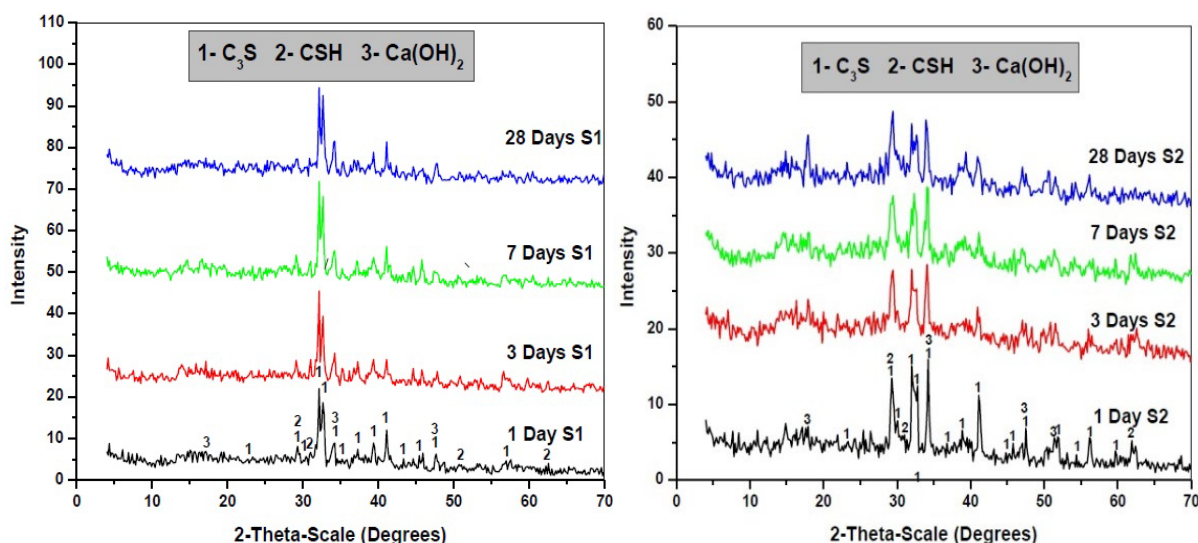


Figure 1: The XRD Plots of Hydrated TCS Paste After 1, 3, 7, and 28 Days, Left for S1 and the Right for S2 [21]

FT-IR of the TCS

TCS is present in different polymorphs and each one has Owen-specific infra-red plots and peaks as shown in Figure 2, so the TCS infra-red spectra 400–4000 cm⁻¹ are mostly caused by the vibration of [SiO₄]⁴⁻ group molecules. There are four vibrational modes in isolated [SiO₄]⁴⁻ tetrahedra, including symmetric stretching vibration V1, asymmetric stretching vibration V3, in-plane bending vibration V2 and out-of plane bending V4. Normally, the four Si-O sp³ hybrid orbits of A [SiO₄]⁴⁻ tetrahedra are completely comparable to those of the equally large numbers of electrons on each track. These electrons are anti-matter, keep clear of each other and maintain a safe distance so that

[SiO₄]⁴⁻ can sustain steady and uniform tetrahedra symmetry. If there is no interference from the outside, the ideal [SiO₄]⁴⁻'s symmetric stretching V1 Tetrahedron has no IR activity [7]. Since the TCS present in different polymorphs, it gives different infrared spectra, but all of them sharing in decreasing in band splitting with more structural symmetry of TCS. The main vibration of pure TCS T1 form is symmetric stretching of the silicate tetrahedra [SiO₄]⁴⁻ V3 at peaks 998, 996, 938, 906, 894, and 883 CM⁻¹, while out-of-plane bending vibration V4 at peaks 525 and 520 CM⁻¹.

The plane bending vibration V2 is present at peaks 464 and 455 CM⁻¹. The symmetric stretching V1 occurs at peaks 846, 834, and 812 CM⁻¹ [7, 15].

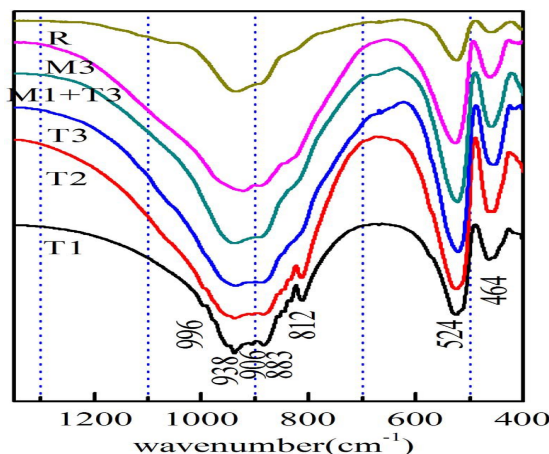


Figure 2: IR Spectra of Different C3S Polymorphs [7]

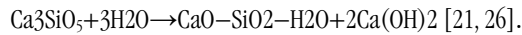
The TCS shows different Infra-Red spectra after hydration (mixing with water). So the peaks 500, 522, 873 CM⁻¹, and 920 CM⁻¹ belong to unreacted TCS (since not all the TCS will react when it will be mixed with water). The peak 968 CM⁻¹ belongs to calcium silicate hydrate gel C-S-H which is the result of the mixing reaction, while the peaks 1417, 1483 CM⁻¹ and 1630, 1643, 3365 CM⁻¹ belong to calcium carbonate CaCO₃ and water respectively. Additionally, the peaks at 1643, 2500, 3400, 3500, 3700 CM⁻¹ belongs to stretching bands of -OH group [15, 21]. The peak related with Ca(OH)₂ and its carbonized products can be

found at 1730 CM⁻¹ [27].

The Effect of Particle Size Reaction of the AND Setting (TCS)

Key qualities of the materials are impacted by the particle size, which is crucial to the hydration process. It has been discovered that inorganic cement made from smaller particle-size powders sets more quickly and has greater compressive strength [15]. When TCS powder is mixed with water, the reacted precipitation process leading to the

development of C-S-H gels, and calcium hydroxide. This reaction can be summarized as below



In the first step, the crystallographic tricalcium silicate react with water. Then C-S-H gel forms, and in the third step, $\text{Ca}(\text{OH})_2$ crystallization takes place. This reaction takes long time which the TCS is unsuitable for dental use that is why commercial TCS dental product contain accelerators like calcium carbonate CaCO_3 which act as a nucleation site for CSH [24], [24,43]. In other product they add calcium chloride CaCl_2 as accelerator [28].

But the above reaction did not happen if the TCS was mixed with resin polymer, namely TheraCal. In this case, although it will never undergo the hydration reaction but still have its merits like calcium ion release, deposition of (HAp), and providing alkaline pH, but to less extent than the hydrated TCS [20].

Other studies also showed that TheraCal provides more Ca ions release than that of ProRoot™ MTA, where the MTA underwent hydration reaction [4].

Bioactivity, Cytotoxicity, and Antibacterial Effect of the TCS

The ability of biomaterials to biologically interact with the bone tissue near them is known as bioactivity. It is often judged by its capacity to produce HAp on its surface when submerged in the simulated body fluid. So, when the TCS is soaked in simulated body fluid, biomaterials made of TCS have the power to trigger the mineralization of hydroxyapatite (HAp). The ability of Osteoblast cells

to develop, differentiate, and express osteogenic genes is significantly influenced by silicon ions obtained from TCS alone or in combination with other compounds.

It has also been used for bone tissue healing. Moreover, it has the ability for inducing cell differentiation and proliferation, as well as having great bioactivity and biocompatibility. Additionally, it has been proved that TCS material is less cytotoxic than glass ionomer cement. The alkaline pH of the TCS, make it act as excellent anti-microbial activity namely *E. faecalis* and *Candida albicans* [15, 28].

Setting of the TCS and its Correlation with Micro-Hardness

When the pure TCS is mixed with an equivalent quantity of water, the setting will occur for 32-36 minutes, but this does not mean that the curing was ceased. At the mixing time not all the TCS will interact with water, there still unreacted TCS, that is why the curing process needs more time up to 28 days. Additionally, we can correlate the curing periods with the micro hardness Vickers values because the Vickers value after 7 days of mixing will increase steadily with progression in time and it will reach maximum value after 28 days after mixing. In dental materials the TCS is not used alone or mixed with water alone. Usually, certain materials were added as modifiers and accelerators to meet the clinical requirements, for example, calcium chloride is added to the liquid of Biodentin as accelerators. So, all these additive materials to TCS may act as setting time determinants.

Table 1 shows some of the additive materials have been added to the TCS and the role of each material in some TCS product available in the dental market [21].

Table 1 (a): The Most Popular Dental Product Depending on TCS Derivatives with their Setting Time, Clinical Use, Composition, and the Roles of the Additive Materials.

Dental Product Depend Mainly on TCS and its Setting Time	Clinical Application of the Dental Product	Composition of Materials	Role of the Additive Material
1-OrthoMTA™ Initial setting time 70 minute. Final setting time 175 minute [28].	1-Root apical plugs and apexification. 2-root perforation. 3-internal resorption. 4-pulp capping [29].	1-Tricalcium silicate, $(\text{CaO})_3\text{SiO}_2$. 2-Dicalcium silicate, $(\text{CaO})_2\text{SiO}_2$. 3-Tricalcium aluminate, $(\text{CaO})_3\text{Al}_2\text{O}_3$. 4-Tetracalcium aluminoferrite, $(\text{CaO})_4\text{Al}_2\text{O}_3\text{Fe}_2\text{O}_3$. 5-Free calcium oxide, CaO . 6-Bismuth oxide, Bi_2O_3 [28, 30].	1-Core material. 2- 2 nd core material. 3-Modifier additive. 4-Modifier additive. 5- Modifier additive. 6-Radiopacifier [28, 30].
2-Bright Endo MTA sealer.™ Initial setting time 15 minute. Final setting time 60 minute [31].	1-general endodontic treatment. 2-surgical endodontic treatment. 3-root perforation. 4-pulp capping. 5-apexification. 6-apicectomy. 7-pulpotomy [31].	1-Calcium silicate CaSiO_4 . 2-Bismuth oxide Bi_2O_3 . 3-Calcium carbonate CaCO_3 . 4- Calcium sulphate CaSO_4 . 5- Calcium Aluminate CaAl_2O_4 [32].	1- core material. 2- Radiopacifier. 3- filler material. 4- Modifier additive. 5- Modifier additive [32].
3- MTA CEM™ 4 week	1-Pulp capping [33]. 2-Root- end filling. 3-Root Repair (Resorption and perforations). 4-Apexification [32].	1-tricalcium silicate. 2-dicalcium silicate. 3-bismuth oxide. 4-calcium. 5-Silicon. 6-aluminium. 7-tricalcium aluminate [34, 35].	1- core material. 2-2 nd core material. 3- Radiopacifier. 4- Modifier additive. 5- Modifier additive. 6- Modifier additive. 7- Modifier additive [34].
4-MTA-Angelus™ 15 minute	1-Root canal perforation and Furcation. 2- Sealing of internal and communicating root resorptions; 3-Retrofilling in endodontic surgeries; 4-Pulp capping in treatments; 5-Pulpotomy; 6-Apexification 7-Apexogenesis [36].	1-tricalcium silicate. 2-dicalcium silicate. 3-bismuth oxide. 4-calcium. 5-Silicon. 6-aluminum. 7-tricalcium aluminate [34].	1- core material. 2-2 nd core material. 3- Radio pacifier. 4- Modifier additive. 5- Modifier additive. 6- Modifier additive. 7- Modifier additive [34].

Table 1 (b): The Most Popular Dental Product Depending on TCS Derivatives with their Setting Time, Clinical Use, Composition, and the Roles of the Additive Materials.

Dental Product Depend Mainly on TCS and its Setting Time	Clinical Application of the Dental Product	Composition of Materials	Role of the Additive Material
5-ProRoot™ Three hours	1- root perforations during root canal therapy; 2-consequence of internal resorption. 3- a pulp capping material. 4-Pulpotomy of primary. 5- root end filling material [37].	1-Tricalcium silicate, (CaO)3-SiO2. 2-Dicalcium silicate, (CaO)2-SiO2. 3-Tricalcium aluminate, (CaO)3-Al2O3. 4-Tetracalcium aluminoferrite, (CaO)4-Al2O3-Fe2O3 5-Gypsum, CaSO4.2H2O. 6-Free calcium oxide, CaO. 7-Bismuth oxide, Bi2O3 [30].	1-core material. 2- 2 nd core material. 3- Modifier additive. 4- Modifier additive. 5- filler material. 6- Modifier additive. 7- Radioopacifier [30].
6-Biodentine™ Initial setting time 6 minute Final setting time 10 minute [28]	1- endodontic repair (root perforations, apexification, resorptive lesions, retrograde filling material in endodontic surgery) 2-pulp capping 3- Dentin replacement material [17]	1-Tricalcium silicate (3CaO.SiO2) 2- Di-calcium silicate (2CaO.SiO2) 3-Calcium carbonate (CaCO2) 4-Zirconium Oxide (ZrO2) 5-Iron oxide [28]	1- core material. 2- 2 nd core material. 3-Filler material. 4-Radioopacifier. 5-colouring agent [28].
7-Theracal LC™ Set by light curing Device [38]	1-direct and indirect pulp capping 2- as a protective liner [38]	1-tri-calcium silicate particles 2-hydrophilic monomer [39]	1-core material. 2-setting agent [39]

Clinical Use of TCS Derivative Products

There are several dental products depending on TCS as main constituents of several dental products that do quite important jobs, table 1. TCS behave friendly with living tissue and enhance cell growth, differentiation, and osteogenic genes expression also induces (HAp) mineralization and deposition. The high PH that acts as antibacterial activity and liberation of Ca ions for enamel remineralization [21, 40-43]. Additionally, it provides maximum adhesion with tooth structure producing zero micro leakage when it used as plug material for root canal perforation [22]. It has been found that TCS derivatives give both clinical and radio graphical success for closer root end and/or immature root formation in permanent teeth particularly it induces proper apical seal with apical healing [23]. The antibacterial effect of this material can be enhanced by adding an extract of the black seed, so the material is compatible with even other additives specifically Black seed extract that enhance the anti-bacterial activity [44].

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Conclusion

Pure TCS material for dental use can be prepared by several methods; namely, solid-state, sole gel method and Pechini Method the behavior of the product concerning the XRD, FTIR, biocompatibility, bioactivity, and antibacterial/antifungal activity antibacterial activity, and particle size, depends on the method of preparation, The Pechini Method produce TCS with the most favorable properties in regards to above properties..

The TCS material is the main constituents of the dental material that are successfully used for several dental therapy namely pulpotomy, root canal sealer, root end treatment and apexification, dentin replacement material, direct pulp capping.

The unique properties and versatility of TCS make them as safe and potential biomaterials that can be used in preventive and restorative dentistry [20].

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