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RESEARCH ARTICLE

NANO-WHITE MTA: A REVIEW.

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Abstract

Mineral trioxide aggregate (MTA) due to its biocompatibility, bioactivity, radio opacity, dimensional stability, insolubility and especially for its unique superior sealing ability has gained wide spread acceptance among clinicians through many years. Due to the drawbacks such as long setting time and poor handling characteristics they are engineered with various constituents to improve their properties. A nano modified version of MTA sets faster with acceptable resistance to acidic environments by adding a small amount of strontium and reducing particle size. High strength and decreased solubility are highly desirable properties for an endodontic root-end filling material. Reducing the size of particles and their uniform distribution has reported to play an important role in shortening the setting time and increasing the microhardness even at a low pH.

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Introduction:-

Biomaterials have been arranged in nature from the micro to the nano-scale and no one can yet combine biological and physical properties to get ideal structures and to create biomimetic structures using smart materials. MTA was developed by Dr.Mahmoud Torabinejad, at Loma Linda University in the 1990s for use as a dental root repair material. MTA has been successfully used in pulp capping, pulpotomy of deciduous and permanent teeth, root end filling procedures, perforation repair, regeneration treatments (Macwan C.et al, 2014).

When MTA powder is mixed with water, calcium hydroxide (CH) and calcium silicate hydrate (C-S-H) are initially formed which are then, transformed into a poorly crystallized and porous solid gel. The ratio of calcium silicate drops because of the formation of a calcium precipitate which produces CH as a byproduct of the hydration reaction. CH is accountable for the biological response of MTA and is the cause of high alkalinity after hydration (Parirokh M. et al, 2010a). MTA is a bioactive material and creates an ideal environment for healing. When MTA is placed in direct contact with the human tissues, it does the following:

1. Forms CH that releases calcium ions for cell attachment and proliferation
2. Creates an antibacterial environment by its alkaline pH.
3. Modulates cytokine production.

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4. Encourages differentiation and migration of hard tissue– producing cells
5. Forms hydroxyapatite on the MTA surface, provides a biologic seal and can also conduct and induct hard tissue formation (Parirokh M. et al 2010b).

Due to the widespread clinical application of MTA, it should possess sufficient radiopacity to be distinct from the adjacent anatomic structures such as bone and tooth. Without a radiopacifier, tricalcium silicate cements have intrinsic radiopacity values ranging from 0.86–2.02 mm aluminium (Al), whereas according to the international standards for dental root canal sealing materials ISO 6876, values lower than 3 mm Al are not recommended. Therefore, a radiopacifier has to be added to tricalcium silicate–based materials. Bismuth oxide is a radiopacifier agent added to MTA in a 1:4 (wt%) ratio, which provides a radiopacity higher than 3 mm Al as suggested by ISO 6876. ProRoot White MTA (WMTA) (Dentsply Tulsa Dental Specialties, Tulsa, OK) has been reported to have a radiopacity ranging from 5.34–6.92 mm Al (Saghiri M.A et al, 2015a).

Currently, MTA is marketed in 2 forms: gray (GMTA) and white (WMTA). MTA was introduced in gray, but because of the discoloration potential of GMTA, WMTA was developed and marketed since 2002. Investigations showed that lower amounts of iron, aluminium, and magnesium are present in WMTA than in GMTA. In endodontics, WMTA (ProRoot, Dentsply Tulsa Dental, Tulsa, OK, USA) has several advantages over other materials used for root repair, including biocompatibility, good sealing ability and antibacterial properties (Saghiri M.A. et al, 2012). GMTA consists of dicalcium and tricalcium silicate and bismuth oxide, while WMTA is primarily composed of tricalcium silicate and bismuth oxide. The WMTA lacks the aluminoferrite phase that imparts the grey colour to GMTA (Parirokh M et al, 2010a). A qualitative surface analysis of WMTA and GMTA showed that the crystal size of GMTA is approximately 8 times larger than that of WMTA. Map images show that oxygen is distributed throughout both crystalline and amorphous phases of GMTA and WMTA, and therefore all of the elements are present in their oxide form (Parirokh M et al, 2010a).

Along with the advantages of biocompatibility and good sealing ability, the drawbacks of MTA include discoloration potential presence of toxic elements in the material composition, difficult handling characteristics, long setting time, high material cost, the absence of a known solvent for this material and the difficulty of its removal after curing.

Malamed reported that in most clinical cases MTA is applied in an inflamed area, which is considered to be an acidic environment. This acidic environment can cause acid corrosion in which CH, C–S–H, and the calcium sulfoaluminate phases decompose and produce porosities. Moreover, MTA becomes considerably porous after its setting time at the MTA–dental interfaces, which results in or causes microleakage (Saghiri M.A. et al, 2014).

In spite of having a better understanding of the material chemistry and recent improvements in physical properties, no material has yet been found that is ideal for any dental application. Attempts have been made to improve the properties of MTA by incorporating materials into its structure by modifying its composition by adding 1% methylcellulose and 2%, 3%, or 5% calcium chloride (CaCl₂) to improve the handling and to lower the setting time of MTA. However, the physical and chemical properties are often affected adversely. Attempts were tried to reduce the particle size of this cement to nano-scales, and adding additives to this composition resulting in a new version of WMTA called the nano-WMTA (NWMTA) (Saghiri M.A et al, 2015b).

Nano-WMTA (NWMTA):-

Nanotechnology is the science of manipulating matter measured in the billionths of meters or nanometer, roughly the size of 2 or 3 atoms

$$10^{-9} \text{ m} = 1 \text{ nm}$$

NWMTA is calcium silicate-based cement composed of nano-sized particles ranging between 40 and 100 nm that provides a surface area about 4 times higher than that of WMTA. An increase in the surface area of the material obtained by nano-sizing the powder particles would (Saghiri M.A et al, 2015b)

1. promote the rise in pH
2. increase the calcium ion release in solution.
3. enhanced the hydration phase of cement particles.
4. better interlocking of powder particles to create a cement structure with better integrity.

It is seen that in mixing and working phase as more particles are involved in reaction with the hydration phase a less porous set material is formed. The difference between the constituent elements of NWMTA and WMTA was related to the presence of strontium with a uniform distribution on the surface. In NWMTA, a constant and uniform nonporous grey image was observed, which might be ascribed to a more proper hydration of products and good interlocking of the crystal compounds of WMTA (Saghiri M.A et al, 2015b). Komabayashi and Spangberg showed that MTA's particle size has a great impact on the extent of particles penetrating the dentinal tubules (Komabayashi T. et al 2008).

NWMTA has been patented by Saghiri et al 2012 (US Patent application No. 13/211.880). (Saghiri M.A. et al 2012)

Table 1:- Ingredients of NWMTA.

Ingredient	Percentage(%)
Disodium hydrogen phosphate	2
Bismuth oxide(bismite)	17
Tricalcium silicate (haturite)	50
Dicalcium silicate (larnite)	15
Calcium sulphate (anhydrite)	2
Gypsum	5
Strontium carbonate	3
Zeolite	2
Tricalcium aluminate	4

The inventors claim that NWMTA exhibits

1. higher resistance acidic environments
2. higher durability in the face of ambient changes
3. better hydration ability
4. better inertness towards toxic ambience
5. better stability with regard to organic and inorganic contaminants
6. better strength
7. less expansion
8. lower porosity
9. improved interfacial microstructure
10. induction of hydroxyapatite formulation
11. better biocompatibility
12. quicker setting time especially in aqueous environment
13. quicker stimulation of hard tissue healing
14. Superior microhardness owing to the presence of NPs
15. A smoother surface that enables better adhesion to the tooth structure & less gaps.
16. Improved cementogenesis
17. Higher osteoconductivity when compared to MTA and Portland cement.

Compared to WMTA, 4 new ingredients have been added to NWMTA namely: tricalcium aluminate, calcium sulphate, zeolite, strontium carbonate

Tricalcium aluminate:-

Used for flash setting. It's use is limited as flash setting is associated with an increase in heat (Saghiri et al 2014).

Strontium carbonate:-

By preventing particle agglomeration, it helps uniform distribution of ingredients. It also reduces setting time and increases the bioactivity of the cement (Saghiri et al, 2012). An energy-dispersive spectroscopy dot map shows that WMTA doesn't have strontium in it's composition. Studies have shown that as a biodegradable material, strontium-doped calcium polyphosphate (SCPP) can release Ca^{2+} and Sr^{2+} as the main degradation products. The release of calcium ion can be important in calcium-mediated signalling during angiogenesis. Strontium salts improve the bioactivity of bone substitute materials (Saghiri M.A. et al 2015b).

Zeolite:- A crystalline hydrated aluminosilicate of alkaline metals and metals of alkaline soils (Ca,K,Na,Mg) is an anticorrosive and stabilising component of NWMTA. It reduces the effect of sulphate attack during the setting reaction (Saghiri et al, 2013a).

Tricalcium aluminate in Portland cement causes an “interstitial phase” which is beneficial due to facilitating the formation of the desired silicate phases. It reacts most strongly with water of all the calcium aluminates, and it is also the most reactive component of the Portland clinker phases. Its hydration to phases of the form $\text{Ca}_2\text{AlO}_3(\text{OH})\cdot n\text{H}_2\text{O}$ leads to the phenomenon of “flash set” (instantaneous set). Due to this advantage, it was reported that tricalcium aluminate is associated with adverse effects that can reduce the durability of concrete by following effects:

Heat release: A large amount of heat is generated during the phenomenon of “flash set” which can cause spontaneous overheating in large masses of concrete. In order to overcome this negative effect first, tricalcium aluminate levels are reduced and secondly small amount of calcium sulfate (typically 4-8%) is added to the cement. Sulfate ions in solution form an insoluble layer of ettringite ($3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 3\text{CaSO}_4\cdot 32\text{H}_2\text{O}$) on the surface of the aluminate crystals, passivize them and slightly contribute to the strength of the cement.

Sulfate attack: The elimination of the sulfate corrosion may be ensured by using low tricalcium sulfate content cement or by adding zeolite to the cement. Zeolite is composed of crystalline hydrated aluminosilicate of alkaline metals and metals of alkaline soils (Ca, K, Na, Mg). Adding zeolite could decrease the volume of tricalcium aluminate in the cement. The amount of zeolite in NWMTA is 2% to prevent its adverse effect on the compressive strength of cement (Saghiri et al, 2014).

Bismuth oxide:-

In MTA, bismuth oxide acts as an inert additive, which does not contribute to the hydration reactions. Previous studies have shown that compressive strength of cements can be negatively affected by bismuth oxide particles. Therefore, nano bismuth oxide was added to NWMTA (Coomaraswamy K.S. et al 2007, Camilleri J.2008). The hydration of CSC begins with the formation of cubic and needle-like crystals (ettringite) in an interlocking orientation in cement structure.

The bismuth oxide particles can affect the hydration process of MTA by their distribution inside the hydrated cement material. Hence, the nano-size bismuth oxide particles, because of their higher surface area, can affect the hydrated structure of cement in a way that the orientation of ettringite crystals can become more uniform, resulting in a dense and solid cement structure. Therefore, nano bismuth oxide enhanced the physical properties of NWMTA.

A study revealed that NWMTA had radiopacity values greater than 3-mm thickness of Al. This implies that they can be candidates for root-end filling materials in terms of their radiopacity. To achieve a material with enhanced physical properties and acceptable radiopacity the ideal percentage for the addition of nano-size bismuth oxide was at least 20% (wt%) (Saghiri M.A. et al, 2015a).

Properties of NWMTA:-

Setting Time:-

Many additives such as CaCl_2 , polymers, plasticizers or other materials have been incorporated to improve setting time. However, they increase toxicity and compromise physical properties. Allen T. confirmed that the surface area of powder is related directly to the setting time of the cement base material (Allen T, 1997).

Final setting time of WMTA is more than 3 h while the initial setting time has been reported to be approximately 40 min (Islam I. et al, 2006) which is not desirable when WMTA is used as a root-end filling material. The initial setting time of NWMTA was approximately 6 min. The difference in initial setting time can be due to the increased surface area of NWMTA than WMTA thereby, NWMTA reacts more rapidly with water and prevents washout of the cement plug before final setting (Saghiri M.A. et al, 2012).

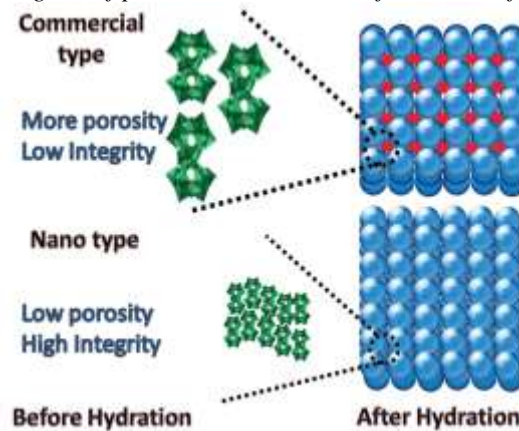
Hydration & Cement dislodgment:-

NWMTA may react more rapidly with water and prevent washout of the cement plug before final setting due to its faster setting time. However, the faster hydration reaction influenced volumetric changes and may lead to dislodgement of cement plug in a clinical scenario (Saghiri M.A., et al 2017).

Push-out bond strength is one of the most important in vitro characteristics that can be extended to a clinical situation and can be used to evaluate the sealing and retentive ability of a root-end filling material and repairing perforation cement during clinical practices. Saghiri et al. reported significantly higher push-out bond strength values for NWMTA compared to WMTA and Bioaggregate (BA) (Saghiri M.A. et al, 2013b). Push-out strength of 138.48 MPa, which is an order of magnitude higher (L. M. Formos et al, 2014).

Hydrated MTA has a weaker microstructure than Portland cement. Additives such as bismuth oxide do not contribute to hydration reactions and ultimately increase the porosity of the cement and decrease the compressive strength of the cement (Coomaraswamy K.S. et al, 2007). In the same manner, tricalcium silicate cement (CSC), with properties similar to those of MTA, showed higher physical properties than CSC with 10% bismuth oxide. It was pointed out that dicalcium silicate in comparison with tricalcium silicate needs more time for hydration which makes MTA require a wet environment in order to gain its optimal physical properties. The effective barrier thickness of this cement is 4mm (Saghiri M.A et al, 2015a).

Figure 1:- Schematic diagram of particles and reaction of cements before and after hydration



NWMTA along with MTA showed superior property in comparison with BA. NWMTA can provide more contact surface area and better hydration which promotes the physical properties of this cement in comparison with BA and MTA. The significant differences between the push-out bond strength values of NWMTA with MTA and BA can be due to the presence of zeolite (Saghiri M.A et al, 2008).

pH:-

Calcium ion content in NWMTA is 80% which is higher than that in WMTA (50-75%). On the other hand, the increase in total surface of NWMTA and improvement in hydration of cement particles leads to an increase in calcium hydroxide formation. Both these phenomenon enhance calcium release from the material and elevate the pH value in the peripheral environment of NWMTA (Saghiri et al 2015 b).

Saghiri et al confirmed that NWMTA containing zeolite had better acidic resistance in comparison with BA or WMTA. Two studies done by Saghiri et al reported that WMTA showed more defects, especially in low pH environments, while NWMTA demonstrated better nucleation of calcium-silicate-hydrated needles (Saghiri M.A. et al, 2013c). Therefore, NWMTA had less porosity, better hydration and good interlocking crystals than WMTA even in an acidic pH. NWMTA kept its high strength in acidic environment compared to WMTA and BA.

Porosity:-

Many studies evaluated the potential for variation in porosity related to the method of manipulation of MTA for condensing the mixed MTA slurry and was concluded that manual hand pressure may produce the least effects on surface porosity (Aminoshariae A.H.G. et al 2003, Nekoofar M.H. et al 2007).

An acidic environment has an adverse effect on the microhardness of WMTA cement (Namazikhah M.S. et al, 2008). WMTA is affected to a large extent than NWMTA, due to the greater porosity of WMTA compared to NWMTA. The greater porosity accelerates acid penetration into the surface texture and decrease surface microhardness. Greater porosity can lead to increased crack propagation. NWMTA has two times less porosity due

to its nanosized particles, leading to reduced crack propagation and acid penetration into the surface texture compared with WMTA (Saghiri M.A. et al, 2012).

Microhardness:-

Microhardness has an inverse relationship with porosity. WMTA with a higher microhardness value has less porosity. For an impermeable root end filling material low porosity is highly desirable. (Saghiri M.A. et al, 2012). Studies have shown that environments with low pH values can adversely affect WMTA by reducing microhardness and increasing microleakage. (Namazikhah M.S. et al 2008 , Saghiri M.A 2008)

At a pH value of 7.4 the microhardness of NWMTA and WMTA were 81.53 ± 2.62 and 51.31 ± 1.14 respectively and in the acidic environment pH= 4.4 the microhardness of both cements decreased to 54.59 ± 1.08 for NWMTA and 16.10 ± 2.84 for WMTA. In acidic environments NWMTA has some cement loss but it has higher microhardness in comparison to WMTA (Saghiri M.A. et al, 2012).

Microhardness, initial setting time and surface porosity comparison of both WMTA and NWMTA revealed significant differences. This might be due to the influence of specific surface area of powders on the exothermic reaction of the cement during hydration, which may affect physical properties of calcium silicate cements to some extent (Saghiri M.A. et al, 2012). The addition of nano-bismuth oxide had a favorable effect on the microhardness of CSCs as it, strengthens the cement by filling the very small areas between hydration products and also enhances the orientation and precipitation of ettringite needles (Saghiri M.A. et al, 2015a)

Compressive Strength:-

In a study, the effect of different pH values on the compressive strength of NWMTA , WMTA and BA was evaluated. The results showed that all tested materials showed their highest compressive strength in alkaline pH (pH=10.4), which were 133.19, 103.63 and 29.07 for NWMTA, WMTA and BA respectively. Lowering the pH to 7.4 decreased the compressive strength of NWMTA, WMTA and BA to 126.81, 86.23 and 25.36 respectively. The lowest compressive strength of tested materials was in acidic pH (pH=4.4). The compressive strengths of 111.41, 63.78 and 10.88 were recorded for NWMTA, WMTA and BA respectively. It was concluded that, NWMTA exhibited the highest compressive strength at all the three pH. When the solubility of WMTA and BA was evaluated under the same pH, the results showed that at a pH value of 10.4, all the tested materials had comparable solubility. However, when the pH value decreased to 7.4, NWMTA exhibited significantly lower solubility compared to BA. At a pH value of 4.4 both Bioaggregate and MTA groups exhibited significantly higher solubility compared to NWMTA.

Therefore, NWMTA showed better compressive strength value in acidic environments compared to WMTA and BA due to less porosity, better hydration, and good interlocking crystals. Therefore, pH alterations can jeopardize the structure of WMTA that may increase/decrease compressive strength (Hashem A.A.R. et al, 2012).

Solubility:-

NWMTA had less solubility than WMTA in low pH. This is due to the characteristics of NWMTA particle form, size, and different composition of this cement. The uniform distribution of particles, and the increased surface area observed in NWMTA, results in less porosity and better interlocking of particles in the set NWMTA and reduced solubility of the cement for a longer time period (Saghiri M.A. et al, 2014). Zeolite, beside anticorrosive action against sulfate, can be regarded as a stabilizing agent for cements. Zeolitic cement can replace Portland cement in many applications with the advantage of higher resistance to acidic and sulfate attack, which could reduce the solubility of the applied cement (Juengsuwattananon K. et al, 2010).

Thermocycling:-

Thermocycling process has been introduced as an artificial aging methodology which is utilized for evaluating the influence of thermal stresses on the bond strength of dental materials. By the means of these thermal cycles in *in vitro* situation, the impact of different coefficients of thermal expansion for these restorative and repair materials can be investigated (Saghiri M.A. et al, 2013b). Investigators have shown that thermocycling regimen comprising a minimum of 500 cycles in water between 5°C and 55°C is an appropriate artificial aging test (The ISO TR1994).

Saghiri M.A. et al evaluated the effects of thermocycling on the push-out bond strength of calcium silicate based cements including WMTA, NWMTA and BA to root dentin and concluded that all three experimental groups showed that thermocycling process had significantly decreased the push-out bond strength of these samples in

comparison with untreated specimens (Saghiri M.A. 2013b). Non-thermocycled NWMTA samples showed cohesive failure modes for the majority of cases which is consistent (Saghiri M.A. et al 2013d, Saghiri M.A.2013b). This type of bond failure can be attributed to the nanoscale particles which are responsible for intimate contact of powder particles that resulted in interlocked pattern and more possibility for cohesive mode of failure. Therefore, thermocycling can adversely affect the push-out bond strength of root-end filling materials. According to SEM images the structural changes made by the thermal stresses from thermocycling process can produce undesirable damages that can decline the dislodgement resistance of these materials. (Saghiri M.A., 2013b).

Biocompatibility:-

Bioactivity is highly desirable for an endodontic root-end filling materials. Recent studies have confirmed that incorporation of a small amount of strontium to bone cements can create or increase bioactivity and bioconductivity properties. (Peng et al, 2010, Yang et al, 2011). Therefore, adding small amounts of strontium (<5%) to WMTA did not adversely affect its physical properties. NWMTA is very biocompatible. As compared to WMTA, NWMTA had better inflammatory response and bone regeneration properties. However, a better bone healing response was seen when nano-tricalcium aluminate was added (Saghiri M.A, et al 2012).

Conclusion:-

Reducing the size of particles and their uniform distribution will better accommodate and fill small gap spaces within the product is reported to play an important role in shortening the setting time and increasing the microhardness even at low pH. NWMTA exhibits higher compressive strength in all pH conditions and can be advocated as an excellent root end filling material, especially when the applied material might be exposed to acidic environments (Saghiri M.A. et al 2013a). Thermal changes can significantly increase the cohesive failure of NWMTA and WMTA which might have negative impact on the sealing characteristics of these cements, especially in the presence of dislodgement forces such as occlusal forces in clinical situation (Saghiri M.A, 2013b).

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