

TO EVALUATE THE MICROCRYSTALLINE STRUCTURE AND ELEMENTAL ANALYSIS OF SET MTA AT DIFFERENT TIME INTERVALS: A SEM STUDY

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ABSTRACT:

Mineral trioxide aggregate is a derivative of Portland cement which was introduced to Dentistry by Torabinejad in early nineties. Its introduction is being considered landmark development as it is used as repair material in most of the pathological conditions of Endodontic origin.

Aim: Evaluation of crystalline surface structure and Elemental of set MTA at different time intervals.

Materials & Methods: 50 Samples of mixed MTA samples were evaluated under Scanning electron microscope at different time intervals. Elemental analysis was also done under EDAX at 24hrs & 72 hrs.

Results: SEM photomicrographs revealed cement surface ranging from crystalline structure with depressions, microchannels to needle like projections. The EDAX revealed the cement to consist of calcium-silicate elements

Keywords: Mineral trioxide aggregate, Scanning electron microscope, EDAX, Alite, Belite, Microchannels.

INTRODUCTION:

Mineral Trioxide Aggregate has been investigated for Endodontic application since early part of 1990's and that was when it was born to literature in dental sciences. Mineral trioxide aggregate (MTA) is a bioactive^[1], crystalline phased mineral powder^[2] which comprises 75% of Portland cement plus 20% of bismuth oxide and 5% of gypsum.^[3] MTA was introduced to dentistry in 1993 by Torabinejad and his colleagues at Loma

Linda University for use as a root-end filling in surgical endodontic treatment.^[4] MTA also contains trace amounts of SiO₂, CaO, MgO, K₂SO₄ and Na₂SO₄.^[5] MTA is prepared as a mixture of powder and water and is used in a slurry form, which gradually hardens in the oral,^[5] wet environment.

A typical analysis of the raw mix, or crude material which enters into the composition of cement and reported as early as 1914 would be as follows^[6]:

Silica-	15.18
Iron alumina oxide	5.06
Calcium carbonate	76.34
Magnesium carbonate	2.90
Undetermined-	.52

While finished cement shows the following analysis ^[6]:

Silica	22.98
Iron alumina oxide	8.80
Lime	63.10
Magnesium oxide	2.42
Sulphuric anhydride	1.42
Loss on ignition	.52
Undetermined	.69

MTA is currently marketed in 2 forms; Gray Mineral Trioxide Aggregate (GMTA) and white Mineral Trioxide Aggregate (WMTA) (Dentsply, Tulsa Dental Products, Tulsa, OK, USA). MTA was introduced in gray colour, but because of the discoloration potential of GMTA, WMTA was developed.^[7] Studies on WMTA revealed that it contains lower amounts of iron, aluminum, and magnesium than in GMTA.^[8-10] GMTA basically consists of dicalcium and tricalcium silicate and bismuth oxide, whereas WMTA is primarily composed of tricalcium silicate and Bismuth oxide^[9]. When MTA powder is mixed with water, calcium hydroxide and calcium silicate hydrate are initially formed and eventually transform into a poorly crystallized and porous solid gel.^[11]

The findings of the first studies were promising and very stimulating with respect to the sealing and repairing properties of this material.^[12] In a short time, MTA gained numerous clinical applications in endodontic procedures and was recommended for direct pulp protection.^[13] MTA has been used as a direct pulp protector and demonstrated remarkable success compared with calcium hydroxide.^[14,15]

MTA has an excellent short and long term biocompatibility^[16] and thus can be used safely adjacent or close to pulp & periodontal tissues.^[13] MTA creates an antibacterial environment by its alkaline pH and encourages differentiation and migration of hard tissue– producing cells.^[17,18,19] The diverse and unique nature of MTA has led its use in many clinical situations where it promises to provide a reliable clinical outcome and long-term prognosis are –

- Pulp capping,
- Lateral root perforations & strip perforations
- Furcation perforation,
- Apical plug in Apexification,
- Root end filling material in Apicoectomy
- Internal and External resorption
- Pulpotomy in primary teeth
- Root canal sealer

MTA has the unique ability to set under moist conditions and has got the property of long setting time. As this cement is being used for various endodontic repair applications it thus becomes imperative to evaluate the set structure of MTA at different time intervals and to understand the role of its various components.

The main aim of this study is-

1. To evaluate the surface crystalline structure of White MTA (WMTA) 24hrs & 72 hrs after it's mixing.
2. To evaluate the elemental analysis of mixed WMTA after 24hrs & 72hrs

MATERIAL AND METHODS:

50 samples were prepared in the group A and 50 samples in group B by using custom made polyethylene cylindrical blocks. Each block had a central hole of 4mm diameter & 6 mm depth. Each sachet of 1 gm WMTA was mixed with 0.33 aliquot of water i.e powder: liquid ratio was 3:1 in accordance with a previous study.^[20] The mixed material was packed incrementally into the cylindrical blocks. The MTA was constantly and evenly compacted in the cylindrical plastic block. The extruded material was removed and a wet cotton gauze was placed on to the MTA. The samples were then stored at 37°C and 95% humidity in an incubator(fig.1). Fifty samples of group A were kept in Incubator for 24hours and the rest fifty samples of group B were kept for 72hours.

Specimens were divided into two groups-

Group A1- 25 specimens in which the surface of MTA was placed in cylindrical block 24hrs after mixing (Fig.2).

Group B1- 25 specimens in which the surface of MTA was placed in cylindrical block 72hrs after mixing (Fig.3).

Samples surfaces of two control group (A 1&B1) were sputter coated with gold and the sample surfaces were analyzed with a Scanning electron microscope (Fig.4). The micrograph images from the SEM analysis of two groups (24h and 72 h) were compared in terms of the surface morphology and type of crystal formation.

Elemental analysis of Group A2 & Group B2 each consisting of 25 samples each was done by EDAX(Energy dispersive analysis of x-rays) to compare the composition at two different timings(24hrs. &72hrs.).

RESULT:

1. SEM Evaluation of set MTA 24hrs after mixing

Observation reveals some depression spots created by air bubbles & micro channels with a smooth cement surface at lower magnification (70x) (fig.5). At a higher magnification of 2500x the crystalline structure seem to consist of few needle like crystals covering the globules scattered between or above the large well defined crystalline plates with the globular particle (alite, belite) (Fig.6)seem to be present on the surface. A few micro channels can be seen embedded between the smaller globules. At 10000x some small crystalline plates look cuboidal or angular in shape while

the larger ones look irregular in shape(fig.7).

2. SEM Evaluation of set MTA 72hrs after mixing

Observations made from the micrograph of unetched MTA 72hrs after mixing reveals more number of depression spots created by air bubbles and a few microchannels at lower magnification (100x), the cement surface looks rough with a more granular surface. At 700x projection from the crystals can be seen on the surface with an amorphous layer covering it with varying no. of microchannels(fig.8). At magnification of 2500x the clustered structure or spiky-ball like clusters looked more evident lying under an amorphous layer with needle-like crystals projecting out on its periphery(fig.9). At 10,000x the surface is smooth with absence of acicular projections but needle like crystals are seen in abundance. The microchannels look more evident with an absence of large cuboidal plate like crystals with increasing magnification .

- 3. EDAX of WMTA 24hrs after its mixing** showed the presence of calcium, bismuth, silicon, magnesium, aluminum, phosphorous, carbon & oxygen. Calcium, bismuth & silicon were the predominant elements. There was no traces sodium present in this group (fig.10).
- 4. EDAX of WMTA 72hrs after its mixing** showed the presence of calcium, bismuth, silicon,

aluminum, phosphorous, carbon & oxygen. Calcium, bismuth & silicon were the predominant elements. There were no traces of sodium & magnesium in this group.

DISCUSSION:

An ideal orthograde or retrograde filling material should seal the pathways of communication between the coronal aperture, root canal system and surrounding tissues. It should also be nontoxic, noncarcinogenic, nongenotoxic, biocompatible with the host tissues, insoluble in tissue fluid. and dimensionally stable.^[21] The diverse applications of MTA have led us to evaluate its surface crystalline structure which has been able enough to create a bond to dentine and cementum thus decimating the chances of leakage during any eventful pathological condition to the tooth.

The inadequately long setting time^[22], porous surface crystalline structure of MTA^[17] and the fact that moistened cotton pallet should be placed in contact with MTA have made researchers believe that many environmental factors may affect the properties of MTA during setting^[23], and a few researchers have offered differing opinions on whether MTA is really superior to other materials in terms of sealing^[24]. It thus becomes imperative to know about the microstructure of MTA and evaluate its components under various physiologic conditions.

The present study has included WMTA (ProRoot MTA. LOT number 09001921)

for its esthetic character and that the use of WMTA with small size particles and finer texture is more advantageous in endodontic treatments in comparison to GMTA^[10]. Custom made Polyethylene tubes that were used in earlier studies^[25,26] were used in this study to pack the MTA incrementally as performed in a previous study by M. B. Kayahan et al^[20].

In the present study, the set MTA surface 24h after mixing showed air voids and micro channels at lower magnification(70x) (Fig.5) while at higher magnification (2500x) microchannels along with globular particles was evident. The globular particles (alite & belite) (Fig.6) a noticeable feature were round in shape consisted of calcium-silicate. This was also depicted by EDAX of the MTA surface 24hrs after mixing which is in accordance with the previous studies^[20,26,27,28]. The peaks in the EDAX of this sample (fig.10) show the elemental dominance of calcium & silicate which proves the fact that MTA is considered as calcium-silicate cement. At 10000x plate like crystals with well defined edges are quite notable and a clear distinction between the small cuboidal crystals and large flat crystals can be made out, with Globules showing definite distinction (fig.7). These findings fall in line with the study by M. H. Nekoofar.^[26] The basic framework of the hydrated mass is formed by the interlocking of cubic and needle-like crystals in which the needle-like crystals form in sharply delineated thick bundles that fill the inter-grain space between the cubic crystals.^[23]

The micrograph of set MTA 72hrs after mixing reveals depression spots or porosities created by air bubbles and a few microchannels at lower magnification (100x& 300x) in accordance with a previous study^[25], the cement surface looks rough with a more granular surface, the presence of air is clearly shown in the EDAX of all the samples of MTA throughout the crystalline and amorphous phases which reveals that all the elements are present in their oxide form.^[10] The porosities can be due to incorporation of microscopic air bubbles during the mixing operation^[4]. 700x magnification shows projecting crystals and a few microchannels (fig.8). At 2500x, the crystals seem to be covered by a blanket of amorphous layer with evenly poised crosssections of micro channels (fig.9). Underneath the amorphous layer the crystals can be seen as spiky-ball like clusters (fig.9) as shown by M. H. Nekoofar.^[26] The clusters seem to be segregated by the crosssections of microchannels. At 10,000x the surface with projected crystals are absent which look more spread and less porous with an increase in the no. of needle-like crystals.

These findings suggest that ettrengite needle like crystals are clearly appreciable only 3days after MTA mixing, while the granular matrix and cuboidal crystals were the main feature of 24h old hydrated MTA, which suggests that presence of ettrengite gets notable with the harder and more set cement, however the EDAX of the two samples (fig.10) does not show any difference between the two groups except than a very minimal increase in

calcium and silicate levels. Absence of FeO in these samples clearly suggests the reason of WMTA acquiring an off-white colour. The elemental analysis of this hydrated MTA done through EDAX are in accordance with previously done studies.^[5,10,28]

Although there are studies relating to the behaviour of MTA in alkaline environment, but there is very limited information about the effect of Acid etching & application of adhesive resin on

the surface crystalline structure of MTA which can indeed decide the nature & timing of an adhesive restoration to be seated on the surface of MTA. Keeping in view the fact that MTA has a prolonged setting time^[22] & is disreputed for its poor handling characteristics,^[29] it becomes all the more important in further studies to know the behavior of hydrated MTA towards its exposure to 37% phosphoric acid followed by application of Adhesive resin to evaluate the bond between MTA and the esthetic restorative materials.

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FIGURES:

Incubator



Fig.1

Set MTA 24hrs. after mixing in the cylindrical block



Fig.2

Set MTA 72hrs. after mixing in the cylindrical block



Fig 3

Environmental Scanning Electron Microscope



Fig. 4

Photomicrograph of Set MTA 24hrs. after mixing shows a) Depression spots created by air bubbles b) Microchannels

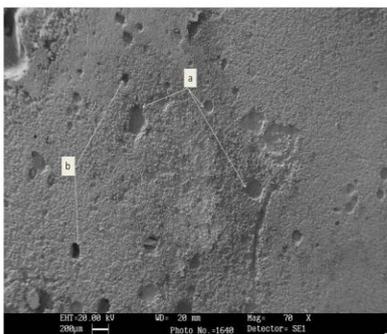


Fig. 5

Photomicrograph of set MTA 24hrs. after mixing shows a) Needle-like crystals(etringite) b) Large well defined crystalline plates c) Globules(alite, belite) d) Microchannels

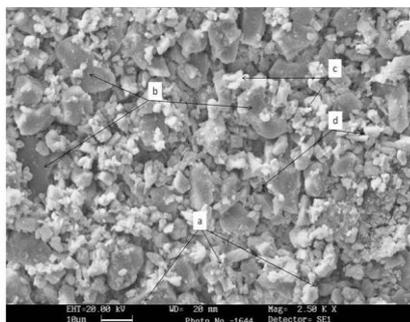


Fig.6

Photomicrograph of Unetched MTA 24hrs. after mixing shows a) Small angular crystalline plates b) large irregular shaped crystalline plates c) Globules

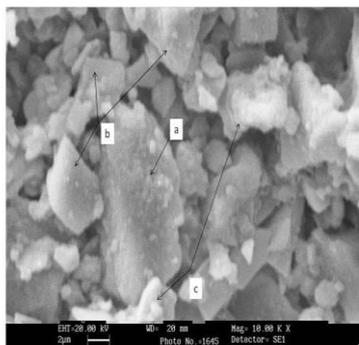


Fig.7

Photomicrograph of Unetched MTA 72hrs. after mixing shows a) Microchannels b) Projecting crystals

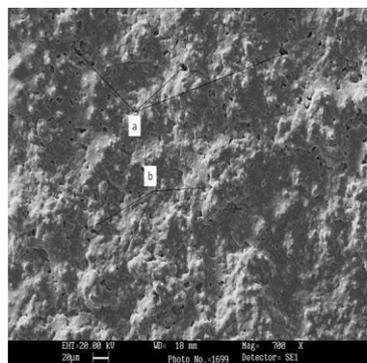


Fig.8

Photomicrograph of Unetched MTA 72hrs. after mixing shows a) Needle-like crystals b) Microchannels c) spiky-ball like crystals and acicular crystals

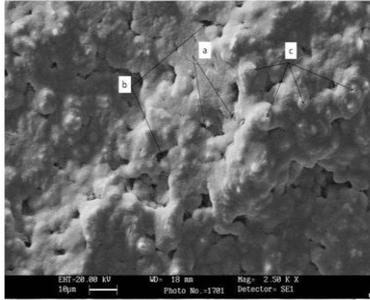


Fig.9

Energy dispersive analysis of x-rays(EDAX) of MTA 24hr. after Mixing

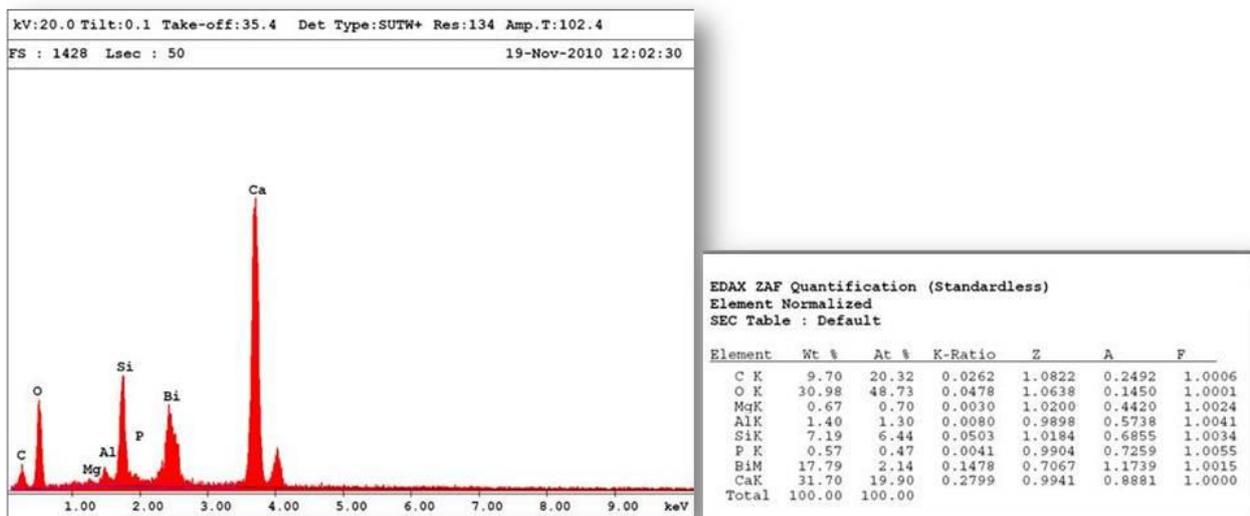


Fig.10