Journal of Applied Pharmaceutical Science Vol. 4 (07), pp. 105-109, July, 2014
Available online at http://www.japsonline.com
DOI: 10.7324/JAPS.2014.40718
ISSN 2231-3354

Comparison of physical & chemical properties of Angelus MTA and new endodontic restorative material

Golsa Salehimehr [1], Shokoufeh Nobahar [2], Seyed-Mostafa Hosseini-Zijoud [3], Somaye Yari [1*]

[1] Endodontic Postgraduate Student, Department of Endodontic, School of Dentistry, Hamadan University of Medical Sciences, Hamadan, Iran. [2] Paediatric Dental Postgraduate Student, Department of Pediatric Dentistry, School of Dentistry, Hamadan University of Medical Science, Hamadan, Iran. [3] Clinical Research Development Unit, Imam Hossein Hospital, Shahid Beheshti University of Medical Sciences, Tehran, Iran.

ARTICLE INFO

Article history:
Received on: 14/05/2014
Revised on: 01/06/2014
Accepted on: 18/06/2014
Available online: 28/07/2014

Key words:
Angelus MTA, New Endodontic Restorative Material, physical properties, chemical properties.

ABSTRACT

In current study the Iranian New Endodontic Restorative Material (NERM) with Angelus MTA, has been compared in term of physical and chemical properties such as; PH, Compressive Strength, release of calcium ions, and final phase of each product and setting time. Five samples of each dental material were prepared based on ISO standards to survey the physical and chemical properties of NERM and Angelus MTA. Structural survey for both dental material revealed a similar crystal structure and no significant differences were observed in the microstructure of the NERM and Angelus MTA. Setting time in NERM and Angelus MTA samples was 25 and 17 minutes, respectively ($P<0.05$). Both materials tested were alkaline and released calcium, the results revealed a higher pH for MTA Angelus than for NERM. The compressive strength values of NERM were greater than the angelus MTA at 14 days. Thus, it can be concluded that NERM has better chemical and physical properties than the Angelus MTA, but Angelus MTA is preferred in terms of setting time. With more studies and considering the physical and chemical properties of NREM, it could be recommended for clinical application because of its accessibility and lower prices.

INTRODUCTION

Various materials in the field of dental science have been formulated, tested and standardized to obtain maximum benefit for good clinical performance. One such new material is Mineral Trioxide Aggregate (MTA), which was developed for use as a dental root repair material by Torabinejad in 1993. MTA was formulated from commercial Portland cement combined with bismuth oxide powder for radiopacity. MTA has been approved by the U.S. Food and Drug Administration in the year 1998. With its numerous exciting clinical applications, MTA promises to be one of the most versatile materials of this century in the field of dentistry (Camilleri and Pitt Ford, 2006). MTA is used for creating an apical plug during apexification, repairing root perforations during root canal therapy and treating internal root resorption and can be used as both a root-end filling material and pulp-capping material. Originally, MTA was dark gray in color, but white versions have been on the market since 2002. MTA contain roughly 20 % bismuth oxide to make the material visible on a dental x-ray (Song *et al*., 2006). A number of previous studies have compared MTA with Portland cement and unequivocally indicated that they are similar in chemical composition, if not identical, except for the inclusion of bismuth oxide in MTA (Asgary *et al*., 2005; Funteas *et al*., 2003).

In vitro (Abdullah *et al*., 2002; Estrela *et al*., 2000) and in vivo (Holland *et al*., 2001; Saidon *et al*., 2003) studies have also shown the Portland cement to exhibit properties similar to MTA. MTA is composed of tricalcium silicate, dicalcium silicate, tricalcium aluminate, tetracalcium aluminoferrite, calcium sulfate and bismuth oxide. Originally, MTA products required a few hours for the initial and final setting, which is uncommon in dental materials. MTA can be a difficult material to use because it is perceived as coarse, sets slowly, and is easily washed out of a moist site (Rao *et al*., 2009).

** Corresponding Author*
Dr. S. Yari , Department of Endodontic, School of Dentistry, Hamadan University of Medical Sciences, Hamadan, Iran.
Email: somayehyari62@yahoo.com

Special delivery systems do not overcome these difficulties. Newer materials are available that set more quickly and have added characteristics. Angelus MTA was introduced in 2001 which contained 80% Portland cement and 20% bismuth oxide. In this restorative material, the calcium sulfate had been removed to reduce the setting time. Angelus MTA is compatible with the human body, has no mutagenic properties, does not cause apoptosis and also has antimicrobial properties and acceptable cytotoxicity (Malhotra *et al*., 2013; Rao *et al*., 2009).

Here is the time to use of materials with desirable properties, therefore designing and manufacturing materials with desirable properties which do not carry Angelus MTA problems is major goal (Kogan *et al*., 2006; Song *et al*., 2006; Wiltbank *et al*., 2007).

Reconstructive products that were produced based on tri-calcium silicate; with different physical and chemical properties have its own advantages and disadvantages. Previous studies have promoted additives to shorten working time, modify MTA's handling properties, and prevent washout (Bortoluzzi *et al*., 2009; Kogan *et al*., 2006).

The new calcium silicate materials have been reported with improved working characteristics (Camilleri, 2008; Kao *et al*., 2009), but evidence is still lacking to support these materials as improvements from MTA. Recently, a new restorative material has been produced as new endodontic restorative material in Iran (NERM) in Iran that partly has the MTA physical and chemical properties.

OBJECTIVES

In current study we decided to compare the new endodontic restorative material in Iran (NERM) with Angelus MTA in terms of physical properties such as setting time, compressive strength, chemical properties such as PH, the release of calcium ions, the final phase of each of these products and the structure of matter.

MATERIALS AND METHODS

In this experimental study, the Angelus MTA (Londrina Brazil) and the New Endodontic Restorative Material (NERM) were prepared. NERM's powder component is consisting of Portland cement, bismuth oxide and its liquid component is consisting of Na_2HPO_4 solution. The Portland cement is fully mixed with bismuth oxide in the ratio of 3 to 1 (i.e., 75 % Portland cement and 25 % bismuth oxide) for preparation of the NERM powder component. The liquid part containing the 0.1 M Na_2HPO_4 solution that is prepared by dissolving the 0.126 g Na_2HPO_4 in 10 mL distilled water. Then 5 samples were prepared based on ISO 2001 68762 standards to check the physical and chemical properties of material (Wiltbank *et al*., 2007).

XRD analysis

The X-ray diffraction (XRD) was used to identify and characterize crystal phases. The prepared sample material was mounted onto the XRD apparatus (Geigerflex Horizontal diffractometer with a graphite crystal monochrometer; Rigaku/MSC, Woodlands, TX). The x-ray beam angle 2_ range was set between 3 degrees (3000) to 70 degrees (70000) and scanned at 2 degrees per minute. The Cu x-ray source was set at accelerating voltage of 45 KV and the current in the electron beam at 30 mA and on continuous scan mode. The peaks on the diffraction pattern were marked using the Rigaku software (version 2.8). Then the peaks were compared and matched with that of the standard material in the powder diffraction file (JCPDS International Center for Diffraction Data 1998, Pennsylvania) using a micro powder diffraction search and matching analysis program.

Microscopic survey

Each sample powder was placed on gold-coated aluminum stop. Analytical scanning electron microscopy was performed on JEOL 6400 SEM (Tokyo, Japan). This microscope was equipped with an Oxford energy dispersive x-ray spectrometer (EDS) and wavelength dispersive x-ray spectrometer (WDS). The EDS system was used to determine the chemical composition of the examined materials.

Setting time

The setting time were determined according to the method described by ASTM C266-03 , which requires the measurement of both initial and final setting times using the initial and final Gillmore needles, respectively. The initial and final setting times of the materials were determined according to these recommendations. The setting times for each material were measured four times. The mean values and standard deviations were recorded for all measurements. Statistical analyses were carried out for setting time using ANOVA and Fisher's LSD at 0.05 level of significance.

pH

The pH of the materials as they set was measured with a pH meter (Orion PerpHect Log R meter, Model 370, Orion Research Inc., Boston, MA) using a temperature-compensated electrode. The readings were taken periodically every 2 min from the start of mixing for 60 min, and then after 24 h. This was repeated three times for each material and the mean pH at each time interval was plotted against time. Statistical analysis was carried out using ANOVA and Fisher's LSD at the 0.05 level of significance at three time points, namely, when the cement was freshly mixed, at 30 min and at 60 min.

Calcium ion release analysis

A total of 5 samples were used for each material. Each tube was sealed in a flask containing 10 mL of distilled water. The amount of calcium released into the deionized water was determined at 5 min, 1 and 24 hours after spatulation. After each measurement, the tubes were moved to new flasks with fresh deionized water. The measurements were performed with the aid of an atomic absorption spectrophotometer (Model GBC 904; CG

Corp, Melbourne, Australia) equipped with a hollow cathode calcium lamp under the following operating conditions: Lamp current: 3 mA, Fuel: acetylene, Support: oxygen, Stoichiometry: reducing, Wavelength: 422.7 nm and Slit: 0.2 nm. To prevent possible interference by phosphates and alkaline metals, all glassware was prewashed with 5% nitric acid. A standard solution of 10 mg/dL of calcium was diluted in 10% EDTA to obtain 0.025, 0.05, 0.1, 0.2, and 0.3 mg/dL concentrations. To calibrate the apparatus for zero absorbency, 10% EDTA was used as the blank. The samples were diluted as necessary to perform the evaluation. The results were calculated by using the equation of the standard curve line.

Compressive Strength

The compressive strengths of the test materials were determined by modifying the method recommended by the BSI . Custommade cylindrical delrin molds 12 mm in length and 6 mm in diameter were used, instead of stainless steel molds as recommended in the BSI, to prepare the specimens for the compressive strength tests. The strength of the materials was determined at 3 days and 28 days after mixing using a Universal Testing Machine (Instron, Model 1334, Instron Corp., Canton, MA). The maximum load required to fracture each specimen was measured and recorded and the compressive strength was calculated in megapascals according to following formula;

$$C=4P/\pi D2$$

where P is the maximum load applied in Newton and D is the mean diameter of the specimen in millimeters. Statistical analysis was carried out using ANOVA and Fisher's LSD at the 0.05 level of significance.

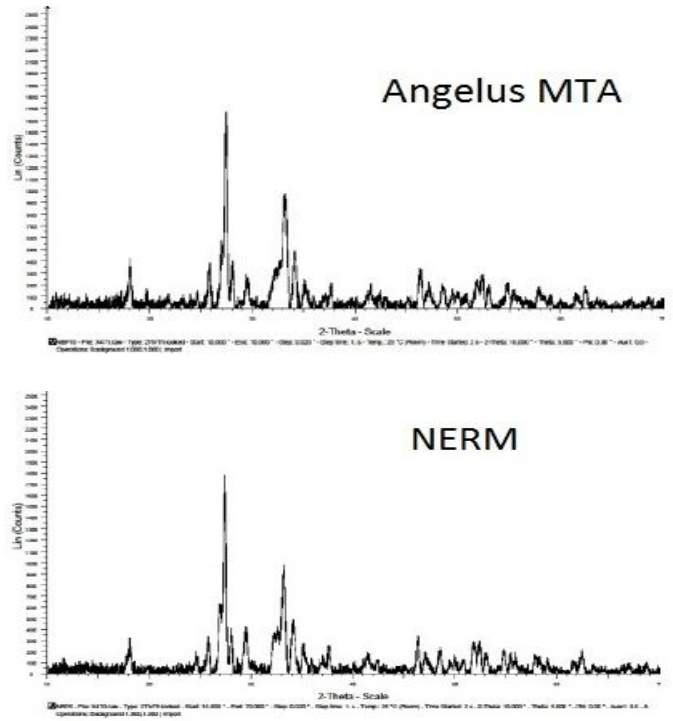

Fig. 1: X-ray diffraction patterns of Angelus MTA and NERM showing peaks representing the crystal phases present in each material.

RESULTS

XRD analysis

The crystal structure of Angelus MTA and NERM were similar (Fig. 1). Both materials were composed mainly of bismuth oxide crystalline structure and calcium silicate oxide. For each of the 2 materials, there were no noticeable differences in the crystalline structure between them.

Microscopic survey

The microstructures of the samples were examined by electron microscopy at three different magnifications. The results showed no significant differences in the microstructure of the two materials (Fig 2).

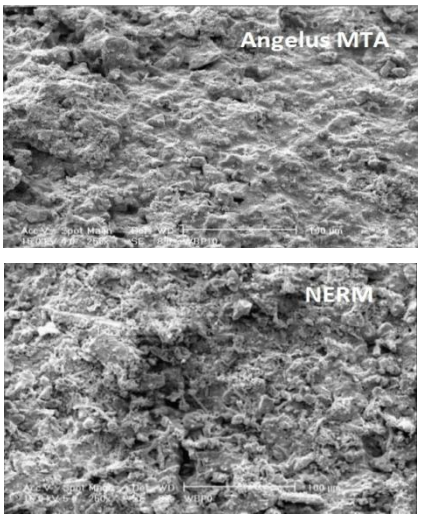

Fig. 2: Microstructure of Angelus MTA and NERM by electron microscopy (X 250).

Setting time

Setting time in NERM samples was about 25 minutes and in Angelus MTA was about 17 minutes ($P<0.05$).

PH

The mean pH values recorded for the materials at the different periods tested are summarized in Table 1. The values for pH were higher during the 5 min, 1 and 24 hours for Angelus MTA when compared with NERM. The pH values recorded for MTA Angelus were higher at all time periods.

Table. 1: pH values recorded at different time periods (mean ± SD).

Materials	5 min	1 hour	24 hours
Angelus MTA	9.46±0.21	10.92±0.74	11.5±0.42
NERM	8.71±0.4	10.01±0.63	11.23±0.71
P value	P<0.01	P<0.01	P=0.5

Calcium ion release analysis

Table 2 presents the mean calcium release at the different time periods. The values for calcium release were higher during the 5 min and 1 hour for NERM, after which it tended to decrease in 24 hours for NERM in comparison with Angelus MTA. Statistically significant difference was reported.

Table. 2: Calcium released (mg/dL) recorded over different periods of time (mean ± SD).

Materials	5 min	1 hour	24 hours
Angelus MTA	0.15±0.03	0.18±0.03	2.14±0.28
NERM	0.3±0.06	0.35±0.11	0.29±0.08
P value	P<0.02	P<0.01	P<0.01

Compressive strength

The compressive strength values of NERM and angelus MTA at different times was shown in fig 3.The compressive strength values of NERM were greater than the angelus MTA at 14 days.

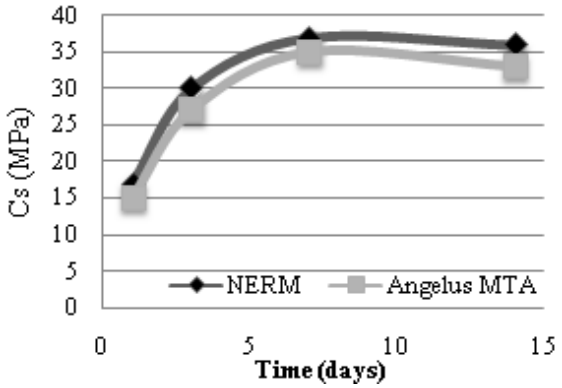

Fig. 3: Compressive strength values of NERM and angelus MTA at different times.

DISCUSSION

Although various types of materials are available for use as a root filling, there is no consensus on choosing the best material as filler. Torabinejad had developed new cement like MTA in 1993 (Torabinejad *et al*., 1995). From then on the formulation of hydraulic calcium (alumina) silicate cements has been studied according to their potential therapeutic use in dental surgery applications (Camilleri and Pitt Ford, 2006; Rao *et al*., 2009).

Portland cement is composed of chemical substances that the tri-calcium silicate, calcium silicate, tri-calcium aluminate,iron tetra-calcium aluminate, and calcium sulfate di- hydrate are the most important ones (Schwartz *et al*., 1999). Portland cement has been used as the main component in the construction of dental material. There are a lot of knowledge and information about Portland cement that can be used to improve and make better dental material (Camilleri, 2008).

MTA is a successful improved sample of Portland cement that could get FDA approval in vivo and in vitro tests and could prove itself as compatible with the human body. Also in NERM samples, the use of phosphate solution as liquid phase can be formed a similar phase and mineral hydroxyl within human body. They can be effective in a better biological adaptation toward Angelus MTA. This theory requires further biological tests (Wiltbank *et al*., 2007). According to the producer of Angelus MTA, this material is composed of 80% Portland cement and 20% bismuth oxide, and no evidence of Calcium Sulfate (Gypsum) is existed in it (Bortoluzzi *et al*., 2009). Results showed that XRD pattern in NERM and Angelus MTA samples are fairly similar, which indicates that these materials have the same crystal structure. Bismuth oxides are added to dental cement due to the characteristics of opaque that are also observed in these compounds. In all cases the formation of calcium silicate hydrate is observed in 29.3, which corresponds to Portland cement hydration and cause increasing the rate of hydration and reducing the setting time (Rao *et al*., 2009; Schwartz *et al*., 1999).

Experiments showed that the present elements (tri-calcium silicate, tri-calcium aluminate and calcium silicate) were very similar to each other and the main ingredient in cement manufacturing and Angelus MTA are equal (Rao *et al*., 2009; Schwartz *et al*., 1999).

Two substances, NERM and Angelus MTA samples, have been evaluated in this study and show the release of calcium ions and PH changes. During the first three hours, its amount is increased and the speed of released calcium ions was rising. According to Angelus MTA samples, in all time intervals, these values were slightly higher and this may be due to large quantities of Portland cement or the release of calcium factors. The achieved alkaline environment is one agent for the treatment of soft and mineral tissue. PH greater than 9 can inactivate the bacteria cell wall. Results indicated that cement produced in an alkaline environment with a PH of less than 12 but Torabinejad reported the higher values (Torabinejad *et al*., 1995).

NERM samples In Figure 2, shows angular and some needle -shaped particles that have surface and are porous. The dimension of these porous particles is about 10 micrometers. The role of networks and porous particles is important during hydration reaction. When the powder is mixed with water, a special structure of the network is created. When the powder and liquid phases were combined in a reasonable amount, they stuck to each other by participating in the gluing process and they became hard at room temperature (Camilleri and Pitt Ford, 2006; Rao *et al*., 2009).

Setting time is one of the important clinical factors, because the prolonged setting time of cement, cause consistency and reduced ability to maintain stability in the shape of cement in oral environment, especially in the presence of solvents. Reduction of setting time makes it difficult to use. The proper setting time is considered as 10 and 15 minutes (Ber *et al*., 2007; Song *et al*., 2006).

Ratio of solid to liquid phase has also effects on the cement strength and setting time. The greater amount of liquid phase cause the less in the viscosity of cement and this has influence on the setting time. The final result is a reduction in strength. Cement setting and hardening reactions take place over time and creates links between hydrate components. Generally setting time of Portland and MTA cement has two stages. First, after mixing the powder and water, the dipping reaction in silicates starts and a gel containing of calcium silicate hydrates is formed and calcium hydroxide is released. In the next step, calcium hydroxide gradually reacts with other minerals and other hydrated compounds are created. Calcium silicate is the main factor in the connection of crystalline calcium hydroxide. Tri-calcium

aluminate plays an important role in the cement setting (Asgary *et al*., 2005; Bortoluzzi *et al*., 2009; Camilleri and Pitt Ford, 2006). NERM and Angelus MTA Samples have setting time of about 25 and 17 minutes, respectively. This result shows that the NERM requires more time for setting.

The compressive strength is not so important for root filling because this type of root filling materials do not bear the direct burden (Oliveira *et al*., 2007). NERM sample stability increases over time and reaches about 35 MPa after 7 days and after that it is relatively fixed in advance.

CONCLUSION

For each of the dental materials, there were no noticeable differences in the composition and crystalline structure between Angelus MTA and NERM samples. The results revealed a higher pH for MTA Angelus than for NERM. NERM setting time and calcium ions release is more than the Angelus MTA. Totally, it can be stated that NERM has better chemical and physical properties than the Angelus MTA, but Angelus MTA is preferred in terms of setting time. With more studies and considering the physical and chemical properties of NREM, it could be recommended for clinical application because of its accessibility and lower prices.

REFERENCES

American Society for Testing and Materials. Standard test method for time and setting of hydraulic-cement paste by Gillmore needles. ASTM C266–03.

British Standards Institution. Specification for Dental Glass Ionomer Cements BS 6039:1981.

Abdullah D, Ford TR, Papaioannou S, Nicholson J, McDonald F. An evaluation of accelerated Portland cement as a restorative material. Biomaterials. 2002; 23(19):4001-10. DOI: 10.1016/S0142-9612(02)00147-3

Asgary S, Parirokh M, Eghbal MJ, Brink F. Chemical differences between white and gray mineral trioxide aggregate. Journal of Endodontics. 2005; 31(2),101-103. DOI: 10.1097/01.DON.0000133156.85164.B2

Ber BS, Hatton JF, Stewart GP. Chemical modification of proroot mta to improve handling characteristics and decrease setting time. Journal of Endodontics 2007; 33(10),1231-1234. DOI:10.1016/j.joen.2007.06.012

Bortoluzzi EA, Broon NJ, Bramante CM, Felippe WT, Tanomaru Filho M, Esberard RM. The influence of calcium chloride on the setting time, solubility, disintegration, and pH of mineral trioxide aggregate and white Portland cement with a radiopacifier. Journal of Endodontics. 2009; 35(4),550-554. DOI: 10.1016/j.joen.2008.12.018

Camilleri J. Modification of mineral trioxide aggregate. Physical and mechanical properties. International Endodontic Journal. 2008; 41(10),843-849. DOI: 10.1111/j.1365-2591.2008.01435.x

Camilleri J, Pitt Ford TR. Mineral trioxide aggregate: a review of the constituents and biological properties of the material. International Endodontic Journal. 2006; 39(10),747-754. DOI: 10.1111/j.1365-2591.2006.01135.x

Estrela C, Bammann LL, Estrela CR, Silva RS, Pecora JD. Antimicrobial and chemical study of MTA, Portland cement, calcium hydroxide paste, Sealapex and Dycal. Brazilian Dental Journal. 2000;11(1),3-9.

Funteas UR, Wallace JA, Fochtman EW. A comparative analysis of Mineral Trioxide Aggregate and Portland cement. Australian Endodontic Journal, 2003;29(1),43-44. DOI: 10.1111/j.1747-4477.2003.tb00498.x

Holland R, de Souza V, Nery MJ, Faraco Junior IM, Bernabe PF, Otoboni Filho JA, Dezan Junior E. Reaction of rat connective tissue to implanted dentin tube filled with mineral trioxide aggregate, Portland cement or calcium hydroxide. Brazilian Dental Journal. 2001; 12(1),3-8.

Kao CT, Shie MY, Huang TH, Ding SJ. Properties of an accelerated mineral trioxide aggregate-like root-end filling material. Journal of Endodontics. 2009; 35(2),239-242. DOI: 10.1016/j.joen.2008.10.023

Kogan P, He J, Glickman GN, Watanabe I. The effects of various additives on setting properties of MTA. Journal of Endodontics. 2006; 32(6), 569-572. DOI: 10.1016/j.joen.2005.08.006

Malhotra N, Agarwal A, Mala K. Mineral trioxide aggregate: a review of physical properties. Compendium of continuing education in dentistry (Jamesburg, NJ : 1995). 2013; 34(2),e25-32.

Oliveira MG, Xavier CB, Demarco FF, Pinheiro AL, Costa AT, Pozza DH. Comparative chemical study of MTA and Portland cements. Brazilian Dental Journal. 2007; 18(1),3-7. DOI: 10.1590/S0103-64402007000100002.

Rao A, Rao A, Shenoy R. Mineral trioxide aggregate--a review. The Journal of Clinical Pediatric Dentistry. 2009; 34(1),1-7.

Saidon J, He J, Zhu Q, Safavi K, Spangberg LS. Cell and tissue reactions to mineral trioxide aggregate and Portland cement. Oral surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontics. 2003;95(4),483-489. DOI: 10.1067/moe.2003.20

Schwartz RS, Mauger M, Clement DJ, Walker WA, 3rd. Mineral trioxide aggregate: a new material for endodontics. Journal of the American Dental Association (1939). 1999; 130(7),967-975.

Song JS, Mante FK, Romanow WJ, Kim S. Chemical analysis of powder and set forms of Portland cement, gray ProRoot MTA, white ProRoot MTA, and gray MTA-Angelus. Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology and Endodontics. 2006;102(6),809-815. DOI: 10.1016/j.tripleo.2005.11.034

Torabinejad M, Hong CU, McDonald F, Pitt Ford TR. Physical and chemical properties of a new root-end filling material. Journal of endodontics. 1995; 21(7),349-353. DOI: 10.1016/s0099-2399(06)80967-2

Wiltbank KB, Schwartz SA, Schindler WG. Effect of selected accelerants on the physical properties of mineral trioxide aggregate and Portland cement. Journal of Endodontics. 2007; 33(10),1235-1238. DOI: 10.1016/j.joen.2007.06.016

How to cite this article:

Golsa Salehimehr, Shokofe Nobahar, Seyed-Mostafa Hosseini-Zijoud, Somaye Yari., Comparison of physical & chemical properties of Angelus MTA and New Endodontic Restorative Material. J App Pharm Sci, 2014; 4 (07): 105-109.