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Investigation of Color Stability and Radiopacity of Novel Tricalcium Silicate Cement, MTA Repair HP.

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Abstract

Introduction: Mineral trioxide aggregate was developed as an apical sealing material, although it has many other indications, such as pulp capping, apexification or perforation sealing. However, some studies have shown that tooth discoloration occurs after the application of WMTA. The radiopacity of MTA depends on the quantity of radiopacifier added. MTA Angelus, a brand of MTA, includes 14 % bismuth oxide. The bismuth oxide in MTA has been implicated in tooth discoloration. A novel material MTA REPAIR HP — "High Plasticity" MTA was recently introduced with the intent to improve their radiopacity and color stability characteristics. A novel material MTA REPAIR HP was recently introduced with the intent to improve some of those characteristics.

Objetive: Evaluate the color stability and radiopacity of MTA Angelus, Repair HP MTA and Biodentine.

Material and Methods: Ten samples of each material were tested under manufacture's conditions. Nine samples were evaluated under free-oxygen conditions and one group control. Radiopacity and color stability was evaluated at the initial setting time, at 5 days, 7 days, 14 days and 30 days.

Results: The MTA Angelus presented dark discoloration and showed statistically significant differences from Biodentine and HP Repair. At the 5 days showed dark discoloration in group MTA Angelus and significantly less discoloration in the other groups. The millimeters of Aluminum in each material have a statistical significant difference between MTA Angelus and HP Repair MTA vs. Biodentine.

Conclusions: MTA Angelus have a higher discoloration in comparation with HP Repair and Biodentine. MTA Angelus have a higher discoloration in the cases with oxygen-free than in the control. MTA Angelus have a higher discoloration during the time exposure after 5 days. There no significant difference between the radiopacity of the MTA Angelus and the HP Repair MTA.

Keywords: Biodentine, bismuth oxide, color stability, white mineral trioxide aggregate, radiopacity, MTA Angelus.

Introduction

Mineral trioxide aggregate (MTA; Dentsply Tulsa Dental Specialties, OK, USA) was developed as an apical sealing material, although it has many other indications, such as pulp capping, apexification or perforation sealing (1,2,3). White MTA (WMTA) was developed for the coronal indications. However, some studies have shown that tooth discoloration occurs after the application of WMTA (4,5,6,7,8).

WMTA is composed of modified Portland cement with added bismuth oxide (9). Bismuth oxide is very effective as a radiopacifying material due to its high atomic weight (10). The radiopacity of MTA depends on the quantity of radiopacifier added. MTA Angelus (Angelus Soluções Odontológicas, Londrina, PR, Brazil), a brand of MTA, includes 14 % bismuth oxide (11) .The bismuth oxide in MTA has been implicated in tooth discoloration (6, 12, 13).

In 2007, Bortoluzzi (14) published a clinical case of coronal and gingival discoloration after repairing a perforation with MTA Angelus. Also, Moore in an in vivo study of apexification in permanent immature incisor with two different types of MTA. The discoloration was present in the 3 months follow-up and at 36 months follow-up.

In 2013, an in vitro analysis with a spectrophotometer demonstrate that there was a coronal discoloration with MTA Angelus and AWMTA (15). Even both induced significant changes in color stability and MTA Angelus was more severe.

There are several conflicts in results regarding the negative effects the bismuth oxide has on the physical properties of the material whereby the strength of the Portland cement is affected by the varying amounts of radiopacifier (16,17). Alternative radiopacifiers have been suggested, thus enabling the replacement of bismuth oxide from the MTA.

Zirconium oxide is used as radiopacifier in Biodentine (Septodont, Saint-Maur-des-Fossés, France) (18). BiodentineTM (Septodont) was developed as a dentin replacement material. Other uses of this material include the restoration of deep and large coronal carious lesions, restoration of deep cervical and radicular lesions, pulp capping and pulpotomy, repair of root perforations, furcation perforations, perforating internal resorptions, external resorption and, apexification and root-end filling in endodontic surgery (19,20,21,22). BiodentineTM is presented as a powder consisting of tricalcium silicate (main component), dicalcium silicate (second main component) and calcium oxide; calcium carbonate (filler material); and zirconium oxide as a radiopacifier. The liquid for mixing with the cement powder consists of calcium chloride (decreases the setting time) and a hydro soluble polymer (water reducing agent) in order to keep a good flowability with a low water/solid ratio (23,24). Biodentine has been shown to be biocompatible (25). BiodentineTM caused the uptake of Ca and Si in the adjacent root canal dentin in the presence of physiological solution (26). There are none references for colour stability for Biodentine.

Other radiopacifying materials have been proposed, such as: iodoform, zinc oxide, lead oxide, bismuth subnitrate, bismuth carbonate and calcium tungstate (27).

A novel material MTA REPAIR HP — "High Plasticity" MTA (Angelus®, Londrina, PR, Brazil) was recently introduced with the intent to improve some of those characteristics (28) . This new formula maintains all the chemical and biological properties of the original MTA; however, it changes its physical properties of manipulation, resulting in greater plasticity, and therefore facilitating manipulation and insertion. Additionally, its formula uses a different radiopacifier calcium tungstate (CaW04) that, according to the manufacturer, does not cause staining of the root or dental crown (29). One of the advantages of the HP Repair MTA is the good sealing for. (29). Up to now, no study evaluated MTA HP push-out bond strength to dentine.

Materials implanted in the human tissues are required to be adequately radiopaque to be distinguished from the surrounding structures so that they can be assessed easily. For these purposes, endodontic materials such as root canal sealers and root-end filling materials include radiopacifying material to enhance the material radiopacity.

The spectrophotometer is an easy instrument that creates different spectral curves with a high precision. It can determinate the color of an object or tooth in 3 different values: L*, a* y b*. There have been described different advantages like the quantification of the color. (30,31,32,33) Other authors used to evaluate the color stability of different materials (34,35) or color dental (36,37)

These three materials, Biodentine, MTA Angelus and HP Repair MTA are retrofilling materials recommended by their biocompatibility and radiopacity. Been accepted such as this material, the smallest radiopacity value accepted for bioceramics by ISO 6876:2001 has established at 3mm (38). According to ANSI/ADS specification number nº57, all bioceramics should be at least 2 mm Al, to improve the radiopacity of dentin or bone (39). Katz et al (40) evaluated the radiopacity of the materials with alongside an aluminum step-wedge, then they compared the grey-scale values with a special software.Nowadays there is not study comparing the colour stability and the radiopacity of MTA Angelus HP Repair MTA and Biodentine. Most of the indications of these bioceramics are in the coronal part, although their colour stability must be with a perfect aesthetic result. For that reason, we decided to check the colour stability in different groups and time-lapses. In this case, we want to test the colour stability of the novel HP repair MTA.

Material and methods

Color Stability Evaluation

Sample Preparation

Ten samples of each material to be tested (Table 1) were mixed in accordance with the manufacturer's instructions. Then with a brusher and a compactor, we placed all the materials in 2 mm × 2 mm silicone tubes. Resulting blocks, we want a perfect setting, under incubator conditions at 37°C at 100% humidity for 48 hours. Two days later, 9 of the 10 samples were immersed in pure glycerin to generate an oxygen-free environment and were irradiated with a Blue phase 20i (Ivoclar-Vivadent, Schaan, Liechtenstein) cure light for 120 seconds. The other one will be part of the control group, with oxygen environment the whole experiment.

Table 1: Materials used in the study

Material	Manufacturer
Angelus Branco	Angelus Soluções Odontológicas, Londrina, PR, Brazil
novel MTA REPAIR HP	Angelus Soluções Odontológicas, Londrina, PR, Brazil
Biodentine	Septodont, St Maure des Fossés, France

Subsequently, one ΔE time intervals were calculated.

Spectrophotometric measurements

Color values were recorded by a single operator using a reflectance spectrophotometer (SpectroShade, Handy

Dental Type 713000; MHT, Arbizzano di Negar, Verona, Italy). After 2 days, after the encourage setting, measurements were taken by positioning the spectrophotometer 2 mm from the samples under constant laboratory light conditions. The instrument was calibrated according to the manufacturer's recommendations before recording the measurements for each group.

Each sample was measured spectrophotometrically at five time points: at 120 seconds of seating, at 5 days ,1 week, 2 weeks, and 1 month after restoration.

The color measurements were reported by using the CIE L*a*b* system. The value of L* is the lightness (from 0 [black] to 100 [white]), the values of a*and b* are the red-green axis and the yellow-blue axis in the chromaticity parameter, respectively. ΔE describes the color difference between the initial time point (after material placement) and each subsequent time point measurement. ΔE was determined by using the following formula:

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{½}$$

Radiopacity evaluation

Radiopacity evaluation of the set materials was performed using ISO 6876:2012 [30] recommendations. Three specimens of each material of 10 ± 1 mm in diameter and 1 ± 0.1 mm thickness. (Table 1) were used. The specimens were radiographed by placing them directly on a photostimulable phosphor (PSP) plate next to a calibrated aluminum step wedge (Everything X-ray Ltd., High Wycombe, UK) with 3 mm increments. A standard Xray machine (GEC Medical Equipment Ltd., Middlesex, UK) was used to irradiate X-rays onto the specimens using an exposure time of 0.16 s at 10 mA, tube voltage at 65 ± 5 kV and a cathode-target film distance of 300 ± 10 mm. The radiographs were processed (Clarimat300, Gendex Dental Systems, Medivance Instruments Ltd., London, UK), and a digital image of the radiograph was obtained. The grey pixel value on the radiograph, of each step in the step wedge, was determined using an imaging program (Adobe Photoshop), and a graph of thickness of aluminum vs. grey pixel value on the radiograph was plotted with the best-fit. The grey pixel values of the cement specimens then determined the relevant thickness of aluminum calculated.

Statistical analysis

For the evaluation of the ΔE, in the Excel Program we introduced all the parameters of each value at different periods of time. A descriptive analysis of the variables was assessed: Percentages of the categoric variables and mean, median and standard deviation for the numeric variables. To assess the effect of oxygen and light on the 3 materials under study, the Δ E values were evaluated if the analysis followed a normal distribution with the Shapiro-Wilks Test. For the possible explanation of the non-normal distribution of the ΔE, a lineal regression model has used in order to evaluate material, time and group variable. After recording data, we performed different graphics in order to evaluate the difference between the cases and control samples, different periods of time (120 seconds, 7 days, 14 days and 30 days).

After confirming a normal distribution for the relation between the type of the material and the millimeters of Aluminum, a x^2 Test were used. If the frequencies were less than 5, we had used the Fisher' contrast. We asses a 95% of confidence interval and the level of significance was set at 0.05.

To express intra- and inter-observer agreement with standard references, kappa was calculated. For the intra-observer agreement, it was a perfect consensus (k=0.90) and a substantial consensus (k=0.77) in the inter-observer agreement.

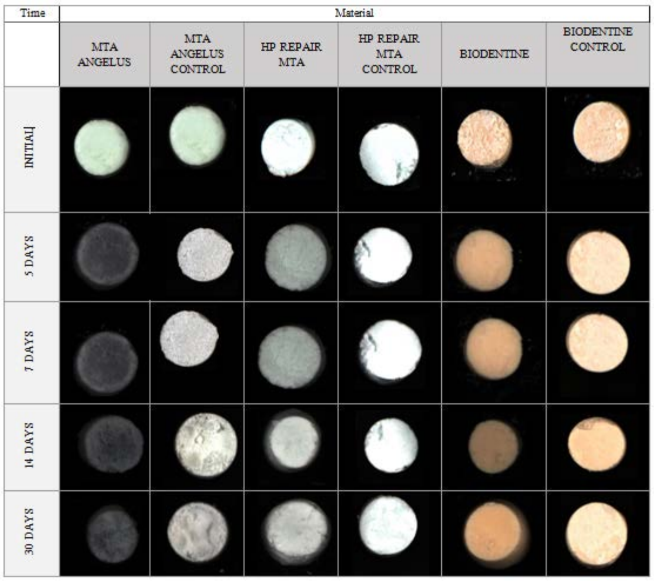

Figure 1: Shows a spectrophotometric image of a sample of each material in the 3 different groups at different time lapses.

Spectrophotometric images from MTA Angelus, HP Repair MTA and Biodentine.

CSM Discoloration According to Oxygen Environment

In the three groups which the group control was in an atmosphere that contained oxygen, all the materials showed color stability over time except MTA Angelus, and there were no significant differences among them (P= 0.086)

In the three groups exposed to Bluephase 20i and fluorescent lamp irradiation, respectively, in an oxygen-free environment with glycerin, the MTA Angelus presented dark discoloration and showed statistically significant differences from Biodentine and HP Repair, which showed less discoloration.

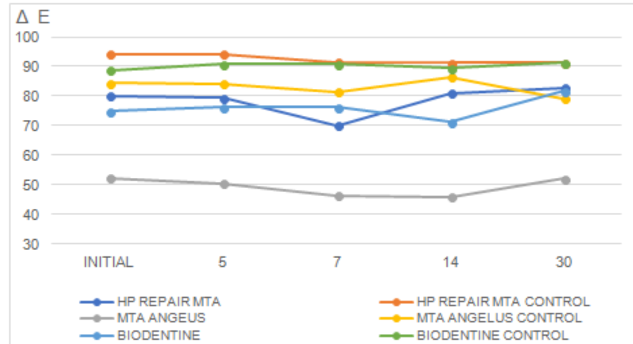

Figure 2: Mean of the cases and control of the materials.

MATERIAL	DAYS									
	INITIAL		5		7		14		30	
	MEAN	SD	MEAN	SD	MEAN	SD	MEAN	SD	MEAN	SD
HP REPAIR	85,65	7,89	81,29	7,32	75,58	3,97	82,53	2,99	82,23	2,00
HP REPAIR CONTROL	94,19	1,54	94,27	1,46	91,58	1,57	91,29	0,19	91,21	0,05
MTA ANGELUS	51,01	3,49	49,09	3,40	45,46	4,21	45,13	3,31	52,22	4,79
MTA ANGELUS CONTROL	84,37	2,83	84,19	3,17	81,33	3,71	86,43	5,10	79,21	0,56
BIODENTINE	75,8	6,63	76,78	6,23	78,97	5,05	73,7	10,65	81,41	1,85
BIODENTINE CONTROL	88,7	1,17	90,76	0,74	90,86	0,90	89,5	0,07	91,21	1,20

Table 2. Δ E values (MEAN+/- SD) of the cases and the value of the controls.

CSM Discoloration According to Exposure Time

Table 2: shows the results of the CSM ΔE evaluation for the 3 groups at 120 seconds of seating, at 5 days,7 days, 21 days and 1 month. At the 5 days showed dark discoloration in group MTA Angelus and significantly less discoloration in the other groups. For evaluate which variables have effect on the ΔE, we adjusted the lineal model with this formula:

$$\Delta E = Alpha + Beta^1 material + Beta^2 group + Beta^3 time + ERROR$$

Lineal Regression Model			
	Estimate Std	P-value	Confidence Value (95%)
MTA Angelus (Intercept) Initial	51,6956	< 0.001 ***	54,401852
Biodentine	24,789	< 0.001 ***	27,2747984
HP Repair	27,2548	< 0.001 ***	29,7406499
MTA Angelus Control	20,5544	< 0.001 ***	23,9371636
T (5 Days)	-0.3395	0.83465	2,8696889
T (7 Days)	-45,88	0.00539 **	5,745178
T (14 Days)	-28,87	0.07748.	-1,378769
T (30 Days)	25,36	0.12049	0,3221556

Table 3: Lineal Regression Model with P-value and Confidence Value 95%. *** Significant Difference.

According to our study MTA Angelus have a statistically significant difference of discoloration comparing with Biodentine and HP Repair MTA (P=< 0.001). Also, with this analysis, there was a significant difference between the MTA Angelus cases and the control (P=< 0.001).

The materials HP REPAIR and MTA Angelus showed no differences between the group control and cases. Biodentine remained stable without discoloration in all the groups over time and showed no significant differences.

Comparing the MTA Angelus at the seating time, there was statistically significant difference at 7 days (P= 0.00539). No significant difference at 5 days, 14 days or 30 days (P= 0.83465, 0.07748, 0.12049).

Radiopacity evaluation

Radiopacity was expressed in millimeters of aluminum equivalent. In order to evaluate the radiopacity we decided to ask 1 blind-evaluators to evaluate from their point of view in how many millimeters of aluminum have each material. To express intra- and inter-observer agreement with standard references, kappa was calculated. For the intra-observer agreement, it was a perfect consensus (*k*=0.90) and a substantial consensus (*k*=0.77) in the inter-observer agreement.

Material	Millimeters of Aluminium			
	0-3 mm	3-6 mm	6-9 mm	9-12 mm
MTA ANGELUS			2	
HP REPAIR		1	1	
BIODENTINE	1	1		

Table 4: Analysis of the millimeters of Aluminum

In our study, we evaluate the radiopacity in two phases: a single observer (A.C.C) evaluate the samples with the Adobe Photoshop. Each material we classify how many millimeters of aluminum have each material. The evaluator (A.C.C) attributed for MTA Angelus from 6-9 mm of aluminum, MTA HP Repair, 6-9 mm and for Biodentine from 0-3mm, almost the beginning from the other stage (3-6 mm).

In order to dismiss this subjective evaluation, we decided to test 1 blind-evaluators with the same procedure. They will only decide which material had the same greyscale from the aluminum stair.

In order to evaluate the relation between kind of material and the mm of Al, we establish the contrast of the x^2 with a P value = <0.001. In conclusion, the millimeters of Aluminum in each material have a statistically significant difference between MTA Angelus and HP Repair MTA vs. Biodentine (P1 P2 <0.001 respectively).

Discussion

Bioceramics are the new material for apical surgery retrofilling, root perforation. Pulp capping and root canals with non-apical stop. It is necessary to create a hermetic obturation, with a successful isolation. Therefore, the material should be selected as well as their properties, such as color stability and staining, if is an anterior tooth or radiopacity if we need a clear differentiation of the surrounding anatomical structures like bone or dentin.

Bortulozzi et al (31) created the first formulation of grey MTA, that provides a limitation in the anterior side due to the discoloration. Later one, white MTA has been introduced in order to eliminate this variation of color, but several studies have shown that also, have discoloration (4). The radiopacifier, bismuth oxide in contact with the tooth structure is the main cause of discoloration. The stability of the bismuth in contact with the oxiding molecules, permits the modification of the material's color (8).

Furthermore, replacement of the radiopacifying agent has been suggested to prevent discoloration (32) They demonstrated that Portland cement does not have sufficient radiopacity to be visualized radiographically and distinguished from adjacent anatomic structures, such as dental tissues and bone. Coomaraswamy et al (14) has shown that when Portland cement got in contact with bismuth oxide the material constants changed by acting as flaws within the cement matrix. This theory demonstrate that bismuth oxide decreased mechanical stability with flaws and increased the porosity with more unreacted water within the Portland cement. These flaws affect the materials solubility and the degradation over time, which affects directly to the longevity.

In our evaluation, MTA Angelus showed dark discoloration if the following conditions were present: an oxygen-free environment, irradiation with a fluorescent lamp, and the presence of bismuth oxide (33).

In the study of Vallés and al (13) concluded that only Biodentine maintain the color stability belong the time compared with CSMs. In their study, they evaluate the color stability in teeth preparation, sectioning the coronal part 1mm below the enamel junction. The contact of the bioceramic with the tooth could be a "key point" of this coloration. According the structure of the tooth, the enamel with this hydroxyapatite component could influence in the evaluation and perception of the discoloration. Another possible explanation was determined by the composition of the different materials (with the chemical reactions with the light-cured). Some manufacturers, such as Medcem GmbH (Weinfelden, Switzerland), claim that better color stability is achieved with PC than with MTA. Lenherr et al compared in bovine tooth model the discoloration when we used endodontics materials. Grey MTA, WMTA and PC have less color stability than PC, and gets stable all the time. Furthermore, when the bismuth was gets in contact with the same PC, the discoloration was developed. Also, Valles et al (34) in a previous study of the stability of Proroot WMTA demonstrate that the formation of metallic bismuth under the irradiation might be the cause of the discoloration of the WMTA samples.

According to Sanz and al (*35*) when we induce a thermal increase of the temperature to the Bi2O3, the number of crystals of bismuth decreased, creating a darkening of the sample. Also, it was observed that crystal melting occurs in noble metal such platinum and gold crucible, not in porcelain. Another possible explanation would be a chemical redox between Pt and Bi2O3. When they increase the melting temperature of Bi2O3 containing glasses produce a color change from pale to deep brown, turning to black at the final phase.

According to this theory, in our study MTA Angelus presented discoloration in an oxygen-free environment and an exposition with light. On the other groups, Biodentine, HP Repair and MTA Angelus control remained a stable in color. In this study, MTA Angelus present the most discoloration followed by HP Repair and Biodentine. This was explained by Camilliari (36) when describe the MTA Angelus with a 10’5% of bismuth oxide. The MTA Repair HP contains calcium tungstate as radio pacifier in the place of bismuth oxide present in earlier products MTA (37-38). Numerous materials have been study such as acid-soluble arsenic levels of Portland cement, MTA Angelus and tricalcium silicate–based. In dental materials there’s only a maximum of 2mg of total arsenic content. Monteiro and Bramante (39) established previous values for This material in MTA Angelus and Portland cement. These levels have been reported such as lowest register. This could be due to the shot time that acid and cement where in contact. In this study the acid where in contact furthermore than 20 minutes, not enough for the complete development. The acid-soluble arsenic must be stand around 16 hours according to the ISO 9917-1 (40)

Regarding the radiopacity, based on the similarity of the aluminum to hydroxyapatite, Goshima et al. (41) decided to design a step wedge because of the linear absorption coefficient. This is the condition that permits the high atomic weight substances determinate their radiopacity. Several studies have evaluated the comparison to an aluminum step wedge with different thickness. Katz et al. (30) establish a comparation of gutta-percha’s radiopacity cones, and the mean radiopacity was around 7 mm Al. Meanwhile, Tanomaru et al (37) evaluated the radiopacity of root canal sealing like AH Plus. Beyer-Olsen and Orstavik (42) determined the radiopacity o root canal sealers using increments of 2 mm with the aluminum step wedge. Their conclusion was that all root sealers were more radiopaque dentin. Shah et and Laghios et al (38) compared different root-end filling materials for differentiate from the radicular dentin and bone with more than 3 mm Al.

Caron G (43) described in their study that Biodentine have a lower radiopacity than MTA Angelus. Meanwhile, a study Galarça et al (44) in radiopacity established that MTA Angelus and MTA HP Repair have almost the same values. In other studies (10) the mean radiopacity for MTA was 7.17mm of thickness of aluminum and Biodentine (30) the mean value was 3,5 mm. There are not several articles than described the physicochemical properties of the MTA HP Repair, therefore, Guimaráes and al (45) concluded that there wasn’t significant difference between conventional MTA and MTA HP Repair ($P < 0.5$) with a 4.5 mm mean of Al thickness. On the other hand, Vivan et al (46) concluded that MTA Angelus had an average of 6,45mm Al thickness. These variations could be due to a change of voltage exposure, or preparation of the sample sizes. Following the manufacture instructions, the mixing ratio of the powder: liquid material should influence in the radiopacity characteristics. A recent study concluded that the more ratio of powder to liquid ratio 4:1 of White MTA, the more radiopacity values were obtained (47).

Porter et al. (48) described a radiopacity of 8,5 mm Al in their study. Otherwise, in their methodology, they established a sample size of 10mm instead of 1mm, which could be an explanation of the higher value of radiopacity according to the theory we have already explain.

Otherwise, not only the radiopacity is an essential characteristic. It has been suggested that the mixture powder to liquid must be respected in order to not modify other properties like physical and biocompatible characteristics. In conclusion, radiopacity is not the only component for a complete success procedure, and more properties should be considered in order to choose the right material.

Evaluating other studies, Baksi et al (49) established that the radiopacity values of the digital analysis technique were lower than those obtained with film radiography. These results of previous studies might be due to the differences among 2 radiographic techniques and their individual technology. There are different parameters that might affect the evaluation of the radiopacity. According to Beyer et al (42) they are changes in exposure time or in milliampere dosage that influence the blackening of the film and no modifications on the contrast. Otherwise, there is no specification of exposure time or milliampere for radiopacity protocols evaluation (29). Moreover, the capacity of absorbance of the aluminum step wedge change with the minimum altering the exposure time and target distance. In their case, also in our study the initial choice of exposure time had no effect on the measured radiopacity for the radiographic method. According to Gu et al (50) an underexposed image decreased the precision of the radiopacity evaluation, creating a low opacity.

In our study, MTA Angelus was the most radiopacifier material comparing with the millimeters of thickness of Al, followed by MTA HPR Repair and Biodentine.

One of the limitations of the study was the quantity of the material. In order to achieve one sample of MTA Angelus or HP Repair Angelus, we must need at least 2 preparations of powder and liquid. Meanwhile, Biodentine with only capsule we achieved finally have 6 samples.

Another limitation for this study was the complexity of the spectrophotometer. The samples with oxygen-free environment, which is mean full of glycerin, suffered disaggregation when the days passed. Then, it was so difficult to have a perfect sample in the whole experiment

Conclusion

According to the results of the present study, MTA Angelus have a higher discoloration in comparation with MTA Repair HP and Biodentine. This might be considered in cases of aesthetic demanding such us anterior side. MTA Angelus also present higher discoloration with oxygen-free condition and after 5 days exposure.

Nevertheless, no significant difference between the radiopacity of the MTA Angelus and de MTA Repair HP.

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