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Influence of Powder-to-Gel Ratio on Physicochemical Properties of a Calcium Silicate Sealer

Influencia de la relación polvo-gel sobre las propiedades fisicoquímicas de un sellador de silicato de calcio

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ABSTRACT: Differences in liquid-to-powder ratio can affect the properties of calcium silicate-based materials. This study assessed the influence of powder-to-gel ratio on physicochemical properties of NeoMTA Plus. Setting time (minutes), flow (mm and mm²), pH (at different periods), radiopacity (mm Al) and solubility (% mass loss) were evaluated using the consistencies for root repair material (NMTAP-RP; 3 scoops of powder to 2 drops of gel) and root canal sealer (NMTAP-SE; 3 scoops of powder to 3 drops of gel), in comparison to Biodentine cement (BIO) and TotalFill BC sealer (TFBC). Statistical analysis was performed using one-way ANOVA and Tukey tests ($\alpha=0.05$). BIO had the shortest setting time, followed by NMTAP-RP and NMTAP-SE. TFBC showed the highest setting time and radiopacity. BIO, NMTAP-RP, and NMTAP-SE had similar radiopacity. All materials promoted an alkaline pH. NMTAP-RP/SE presented lower solubility than BIO and TFBC. Regarding the flow, TFBC had the highest values, followed by NMTAP-SE, and NMTAP-RP. BIO had the lowest flow. In conclusion, NMTAP in both powder-to-gel ratios showed high pH and low solubility. The increase in the powder ratio decreased the setting time and flow. These findings are important regarding the proper consistency and work time to clinical application.

KEYWORDS: Calcium silicate; Dental materials; Endodontics; Physicochemical properties; Root canal sealer; Root repair material.

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RESUMEN: Las diferencias en la proporción líquido/polvo pueden afectar las propiedades de los materiales a base de silicato de calcio. Este estudio evaluó la influencia de la proporción polvo/gel en las propiedades fisicoquímicas del cemento NeoMTA Plus. El tiempo de fraguado (minutos), la fluidez (mm y mm²), el pH (en diferentes períodos), la radiopacidad (mmAl) y la solubilidad (% de pérdida de masa) fueron evaluados utilizando las consistencias para el material de reparación radicular (NMTAP-RP; 3 cucharadas de polvo/2 gotas de gel) y para cemento sellador del conducto radicular (NMTAP-SE; 3 cucharadas de polvo/3 gotas de gel), en comparación con el cemento Biodentine (BIO) y el cemento TotalFill BC (TFBC). El análisis estadístico se realizó utilizando las pruebas ANOVA y Tukey unidireccionales (α=0.05). BIO tuvo el tiempo de fraguado más corto, seguido de NMTAP-RP y NMTAP-SE. TFBC mostró el mayor tiempo de fraguado y radiopacidad. BIO, NMTAP-RP y NMTAP-SE tuvieron una radiopacidad similar. Todos los materiales promovieron un pH alcalino. NMTAP-RP/SE tuvieron una solubilidad menor que BIO y TFBC. Con respecto a la fluidez, TFBC tuvo los valores más altos, seguido de NMTAP-SE y NMTAP-RP. BIO tuvo la fluidez más baja. En conclusión, NMTAP en la relación polvo/gel mostró un pH alto y una baja solubilidad. El aumento en la proporción de polvo disminuyó el tiempo de fraguado y la fluidez. Estos hallazgos son importantes con respecto a su consistencia y tiempo de trabajo durante la aplicación clínica.

PALABRAS CLAVE: Silicato de calcio; Materiales dentales; Endodoncia; Propiedades fisicoquímicas; Cemento sellador del conducto radicular; Material de reparación radicular.

INTRODUCTION

The development of calcium silicate-based materials represents one of the most important advances in reparative dentistry and Endodontics (1). In Endodontics, these materials are indicated for vital pulp treatment, root-end filling, sealing of perforations, and regenerative procedures (2), due to their interaction with dentin, promoting a biomineralization process at the cement-dentin interface (3).

MTA Plus (Avalon Biomedic Inc. Bradenton, USA) is a calcium silicate-based material developed with smaller particles than MTA (4), which provide an increase in the hydration of cement surface, higher resistance and better handling (5). NeoMTA Plus (Avalon Biomedic Inc. Bradenton, USA) was later developed with tantalum oxide radiopacifier to replace bismuth oxide (6) to prevent interference in cement hydration and dental discoloration (7). Neo MTA Plus presents ability to release calcium and phosphate ions contributing to hydroxyapatite deposition (8), and bioactivity (9,10). This material contains powder and a water-based gel. NeoMTA Plus is indicated for root canal filling or as a repair cement depending on its consistency. When the proportion of gel is increased, a sealer consistency is obtained (11).

Biodentine (Septodont, France) is another material based on calcium silicate developed to overcome some disadvantages reported for MTA (2). Biodentine has short setting time, high mechanical strength, and superior handling property than MTA (12). Furthermore, Biodentine has zirconium oxide as radiopacifier (13), and presents high biomineralization capacity than NeoMTA Plus (10). Endosequence BC (Brasseler USA, Savannah, GA) and TotalFill BC (FKG Dentaire SA, La Chaux-de-Fonds, Switzerland) are premixed, ready-to-use calcium silicate materials (14) including a putty, indicated as root repair material and a sealer to root canal obturation (15). BC Sealer hydrate by contact with environmental moisture (15), and presents an alkaline pH, flow, film thickness and radiopacity according to ISO 6876 (16-18).

Previous studies have shown that the powder-liquid ratio for manipulation of endodontic materials influences their physicochemical properties (19, 20). However, there are no studies evaluating the influence of powder-to-gel ratio on physicochemical properties of NeoMTA Plus. Therefore, the aim of this study was to evaluate the setting time, pH, solubility, radiopacity and flow of NeoMTA Plus using the consistencies for repair material (NMTAP-RP) and root canal sealer (NMTAP-SE), in comparison with Biodentine cement (BIO) and TotalFill BC Sealer (TFBC).

MATERIALS AND METHODS

The endodontic materials used in this study and their respective composition, proportion, and manufacturers are described in Table 1.

Table 1. Endodontic materials, their manufacturers, compositions, and proportions used.

Material	Manufacturer/Composition	Proportion
NeoMTA Plus (NMTAP-SE)	Avalon Biomed Inc, Bradenton, FL / Powder: tricalcium silicate, dicalcium silicate, tantalum oxide, tricalcium aluminate, and calcium sulphate. Liquid: water-based gel with thickening agent and water-soluble polymers	1 g powder: 0.45 mL gel/ 3 scoops of powder to 3 drops of gel
NeoMTA Plus (NMTAP-RP)	Avalon Biomed Inc, Bradenton, FL / Powder: tricalcium silicate, dicalcium silicate, tantalum oxide, tricalcium aluminate, and calcium sulphate. Liquid: water-based gel with thickening agent and water-soluble polymers	1 g powder: 0.30 mL gel/ 3 scoops of powder to 2 drops of gel
TotalFill BC Sealer (TFBC)	FKG Dentaire SA, La Chaux de Fonds, Switzerland / Zirconium oxide, calcium silicates, calcium phosphate monobasic, calcium hydroxide, filler and thickening agents	Single syringe Ready-for-use
Biodentine (BIO)	Septodont, Saint-Maur-des-Fossés, France / Powder: tricalcium silicate, calcium carbonate, zirconium oxide, dicalcium silicate, calcium oxide, iron oxide. Liquid: aqueous solution of a hydrosoluble polymer with calcium chloride	1 g powder: 0.34 mL liquid/ 1 capsule of powder to 6 drops of liquid

SETTING TIME

Type IV plaster molds (Durone IV Salmon, Dentsply, Petrópolis, RJ, Brazil) measuring 10 mm in diameter and 1 mm high were fabricated (n=6) and immersed in distilled water for 24 hours at 37ºC. The molds were filled with the materials. Setting time was evaluated in accordance with the ISO 6876:2012. A Gilmore needle with mass of 100 g and diameter of 2 mm was used, supported on the material surface. The materials were kept in an oven at 37ºC and 95% humidity between the measurements. The setting time of the cements was considered as the time when the marks of needle could not be observed on the material surface.

PH

Polyethylene tubes (Embramed Ind. Com., São Paulo, SP, Brazil) measuring 10 mm high by 1 mm diameter were filled with freshly prepared samples of each material (n=10). Each tube was placed into a separate flask, containing 10 mL distilled water. The samples were stored at 37°C, and pH measurement was performed after incubation of 12 hours, 1, 7, 14, and 21 days. The solutions pH was analyzed at each period using a previously calibrated digital pH meter (Digimed, SP, Brazil). The control was based on the pH values of distilled and deionized water.

SOLUBILITY

This test was based on a previous study (21). Circular plastic molds with an internal diameter of 7.5 mm and a height of 1.5 mm were used for sample preparation. These molds were filled with the evaluated materials and a nylon thread (n = 6). A glass plate was placed on top of the molds, exerting a light pressure in order to remove any excess. As the materials require moisture for setting, 2 pieces of wet cloth were placed between the mold and the glass plates (14). All samples were kept at 37°C and 95% humidity for three times the duration of their setting time, as stablished in the setting time evaluation. The test specimens were removed from the molds and weighed on a precision balance (Adventurer AR2140, Ohaus Corporation USA. Toledo do Brasil Indústria de Balanças Ltda., São Bernardo do Campo, SP, Brazil). The specimens of each material were individually placed in plastic flasks, containing 7.5 mL of distilled water. The flasks were then transferred to an oven at 37°C where they remained for 7 days. After this period, they were washed in distilled water, and placed in a dehumidifier. The mass was measured before and after the samples were immersed in distilled water, and every 24 h thereafter, until the mass was stabilized. The loss of mass was expressed as a percentage of the original mass.

RADIOPACITY

Specimens (n=6) measuring 10 mm in diameter by 1 mm thickness were made for each tested material. As the materials require moisture for setting, 2 pieces of wet cloth were placed between the mold and the glass plates (14). The specimens were kept in an oven at 37°C and 95% relative humidity for three times the duration of their setting time. Each sample was positioned on occlusal radiographic films (Insight-Kodak Comp, Rochester, NY, USA) and exposed, along with an aluminum stepwedge with variable thickness (from 2 to 16 mm, in 2-mm increments). An X-ray unit (Instrumentarium Dental, Tuusula, Finland) operating at 60 kV, 7 mA, 0.32 pulses per second, and focus-film distance of 33 cm was used. The films were processed in a standard automatic processor (Dent-X 9000, Dent-X, Elmsford, USA). Radiographs were digitized, and the images were imported to the Image Tool 3.0 software (UTHSCSA, San Antonio, TX, USA); an equal-density tool was used to identify equal-density areas in the radiographic images. Thus, the radiopacity of the evaluated sealers was estimated from the thickness of aluminium (in mm) by using a conversion equation. The values recorded for each material were averaged to obtain a single value in mm Al.

FLOW

The flow test was conducted based on ISO Standard 6876:2012. 0.05 mL of the materials was placed in the centre of a glass plate using a graduated syringe (n=10). After manipulation, another glass plate (20 g) was placed on the plate with the sealer, and a 100-gram weight was put on the top plate, and kept there for 10 minutes. After this period, the maximum and minimum diameters of the material on the glass plate were measured. When a difference of less than 1 mm between the diameters was observed, the mean value was recorded. A second evaluation was made by photographing the material on the plate alongside a millimetre ruler. The images obtained were evaluated using the Image Tool version 3.0 program to obtain the area of flow of the material expressed in mm^2.

STATISTICAL ANALYSIS

All data were analyzed with the GraphPad Prism 7.00 (GraphPad Software, La Jolla, CA, USA) statistical software package. The normality of the data distribution was confirmed with a Shapiro-Wilk test, and the data were analyzed by one-way analysis of variance and Tukey multiple comparison tests. The level of significance was accepted as $P<0.05$.

RESULTS

Setting time, solubility, radiopacity and flow are described in Table 2. All the materials had significant differences regarding the setting time. NMTAP-SE and NMTAP-RP showed greater setting time than BIO and lower than TFBC ($P<0.05$). Regarding solubility, NMTAP-SE and NMTAP-RP had the lowest values, with no difference between the two consistencies ($P>0.05$). BIO and TFBC had solubility higher than the recommended by ISO 6876 ($P>0.05$). TFBC presented the highest radiopacity ($P<0.05$). There was no difference between the other materials ($P>0.05$). When evaluating flow, TFBC had the highest values, followed by NMTAP SE, NMTAP RP and BIO ($P<0.05$).

The pH values in the different periods are shown in Table 3. All the cements had alkaline pH after 12 hours. TFBC presented the highest pH values ($P<0.05$), followed by NMTP-RP and NMTAP-SE ($P>0.05$). BIO had the lowest values after 12 hours, 1, 7 and 14 days, with higher values than the control group (distilled and deionized water) ($P< 0.05$). After 7 and 14 days there was no statistical difference among NMTAP-RP, NMTAP-SE and TFBC ($P>0.05$). After 21 days there was no difference among groups ($P>0.05$).

Table 2. Solubility, setting time, radiopacity and flow values (Mean and Standard Deviation).

Material /Test	NMTAP-RP	NMTAP-SE	TFBC	BIO
Solubility (% mass loss)	0,51 (±0,14)^{a}	1,13 (±0,19)^{a}	7,21 (±0,55)^{b}	6,51 (±0,35)^{b}
Setting time (minutes)	87,67 (±5,54)^{b}	238,00 (±13,77)^{c}	560,90 (±29,18)^{d}	26,33 (±1,37)^{a}
Radiopacity (mm Al)	2,85 (±0,21)^{b}	2,45 (±0,19)^{b}	6,36 (±0,62)^{a}	2,78 (±0,29)^{b}
Flow (mm)	8,78 (±1,32)^{c}	13,98 (±1,25)^{b}	22,95 (±0,55)^{a}	6,79 (±0,33)^{d}
Flow (mm^2)	64,88 (±17,63)^{c}	183,40 (±31,26)^{b}	515,70 (±50,75)^{a}	46,74 (±4,21)^{d}

Different superscript letters represent statistically significant difference between the materials. Statistically significant at $P<0.05$.

Table 3. pH values (mean and standard deviation) at the different experimental periods.

Material /Periods	Control (H2O)	NMTAP-RP	NMTAP-SE	TFBC	BIO
12 hours	6,39 (±0,35)[d]	10,96 (±0,22)[b]	10,73 (±0,14)[b]	11,43 (±0,27)[a]	9,26 (±0,19)[c]
1 day	6,99 (±0,25)[d]	9,82 (±0,26)[b]	10,09 (±0,43)[ab]	10,55 (±0,12)[a]	8,61 (±0,0)[c]
7 days	6,47 (±0,25)[c]	10,50 (±0,37)[a]	10,32 ±0,51)[a]	10,32 (±0,11)[a]	9,62 (±053)[b]
14 days	6,37 (±0,26)[c]	10,68 (±0,13)[a]	10,49 (±0,43)[a]	10,55 (±0,12)[a]	9,79 (±0,56)[b]
21 days	6,58 (±0,34)[b]	10,16 (±0,56)[a]	10,06 (±0,62)[a]	9,55 (±0,98)[a]	10,16 (±0,39)[a]

Different superscript letters represent statistically significant difference among the materials. Statistically significant at $P<0.05$.

DISCUSSION

New calcium silicate-based materials have been developed (2,22). Therefore, it is important to investigate their performance in different clinical applications (8). The present study is clinically relevant since suitable material properties must be obtained when different ratio powder/gel is used for proper consistency as reparative material or endodontic sealer, which are indications of NeoMTA Plus (19).

A short setting time of endodontic materials decreases the risk of displacement and contamination (23). Biodentine had the shortest setting time, probably due to the presence of calcium chloride in its composition (13). Following Biodentine, NeoMTA Plus showed the lower values and the increase in the powder ratio decreased its setting time. The water-based gel present in NeoMTA Plus contains accelerators for setting, justifying the present results (9). TotalFill BC Sealer had the longest setting time, corroborating previous studies (14,16, 8).

Endodontic materials have contact with body fluids. Thus, it is important to determine the solubility of the materials in order to evaluate their stability (12). Fridland & Rosado (20) evaluated the solubility and porosity of Pro Root MTA (Tulsa Dental, OK, USA) using different water-to-powder ratios. The authors showed that both solubility and porosity increased when the amount of water was increased. In agreement, Cavenago *et al* (19) also observed greater solubility for White MTA (Angelus, PR, Brazil) when the amount of water was increased. On the other hand, our results showed that NeoMTA Plus in both consistencies had a similar low solubility. This result may be associated to its shorter final setting time, when compared with the conventional MTA (24), besides its hydration mechanism and setting expansion (25).

Even with a short setting time, Biodentine had a high solubility, as previously reported (25). This high solubility probably occurs due to the presence of polycarboxylate in Biodentine, a hydrosoluble polymer that has a surfactant effect and may disperse the cement particles (26). TotalFill BC Sealer had the highest values of mass loss, corroborating a previous study (14). One reason for that is the hydrophilic nanosized particles of this sealer, which increase their surface area and allows more liquid molecules to come into contact with the sealer (27).

Calcium silicate materials produce an alkaline pH, due to the release of calcium ions (28), and the repair mechanism of mineralized tissue depends on the pH and $Ca^{2}+$ release capacity of the material (29). All the materials evaluated had high values of pH, in agreement with previous studies (14,18,25).

It is recommended that the endodontic materials exhibit a radiopacity of at least 3 mm Al. The differences on radiopacity of the materials evaluated can be justified by the presence of the different radiopacifying agents of each material in addition to their amount and proportion (30). NeoMTA Plus presents tantalum oxide as a radiopacifying agent, whereas TotalFill BC Sealer and Biodentine have zirconium oxide. TotalFill BC Sealer had higher radiopacity than the other materials. NeoMTA Plus and Biodentine had similar values to each other, being smaller than 3 mm Al, in agreement with previous studies for Biodentine (31,32). This a reflection from the composition of Biodentine, which has only 5% of zirconium oxide (13). The consistence of NMTAP didn't interfere in its radiopacity.

Sealing ability may be related to the flow of endodontic materials (33). TotalFill BC Sealer had the highest and Biodentine the lowest flow rate, in agreement with other studies (14,17,34). There is no study evaluating the flow of NeoMTA Plus. Although our results have shown that NeoMTA Plus did not comply with the ISO standards, McMichael *et al* (11) observed a high penetrability for this material. One explanation for that could be the absence of association between flow and filling properties (34).

A limitation of the current study is that the tests based on the ISO 6876 standard may not be ideal to evaluate hydraulic materials (35,36), and the results from *in vitro* studies should be carefully interpreted in comparison to a clinical condition (37). Nevertheless, these *in vitro* results contribute to the planning of *in vivo* and clinical investigations (38).

CONCLUSION

In conclusion, NMTAP in both powder-to-gel ratio showed high pH and low solubility. The increase in the powder ratio decreased its setting time and flow. These findings are important regarding the proper consistency and work time to clinical application.

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