This study determined the chemical composition, pH, and radiopacity of mineral trioxide aggregate (MTA), and also compared the setting time, compressive strength, and solubility of this material with those of amalgam, Super-EBA, and Intermediate Restorative Material (IRM). X-ray energy dispersive spectrometer in conjunction with the scanning electron microscope were used to determine the composition of MTA, and the pH value of MTA was assessed with a pH meter using a temperature-compensated electrode. The radiopacity of MTA was determined according to the method described by the International Organization for Standardization. The setting time and compressive strength of these materials were determined according to methods recommended by the British Standards Institution. The degree of solubility of the materials was assessed according to modified American Dental Association specifications. The results showed that the main molecules present in MTA are calcium and phosphorous ions. In addition, MTA has a pH of 10.2 initially, which rises to 12.5 three hours after mixing. MTA is more radiopaque than Super-EBA and IRM. Amalgam had the shortest setting time (4 min) and MTA the longest (2 h 45 min). At 24 h MTA had the lowest compressive strength (40 MPa) among the materials, but it increased after 21 days to 67 MPa. Finally, except for IRM, none of the materials tested showed any solubility under the conditions of this study.

When nonsurgical root canal therapy fails or cannot be performed, surgical root canal therapy is conducted. The procedure usually consists of root-end exposure and resection, as well as preparation of a class I cavity and placement of a root-end filling material. A number of substances have been suggested and used as root-end filling materials (1).

Recently, an experimental substance, mineral trioxide aggregate (MTA), has been suggested as a potential root-end filling material. In a series of in vitro studies, Torabinejad et al. (2-5) evaluated the sealing ability of MTA, compared with commonly used root-end filling materials. Statistical analysis of their data showed that MTA had significantly less dye (2, 3) and bacterial (4) leakage than amalgam, Super-EBA or Intermediate Restorative Material (IRM). In addition, when marginal adaptation of MTA root-end fillings was compared with those of amalgam, Super-EBA, and IRM under scanning electron microscope, they reported no noticeable gap between MTA and its surrounding dentinal walls (5).

Because these studies show that MTA leaks less and has better adaptation to the root-end cavity walls than commonly used root-end filling materials, its other characteristics—such as chemical composition, physical properties, antibacterial effects, cytotoxicity, and biocompatibility—should also be investigated.

The purpose of this study was first to determine the chemical composition, pH of the setting cement, and radiopacity of MTA, and second to compare the setting time, compressive strength, and solubility of this material with those of three commonly used root-end filling materials, amalgam, Super-EBA, and IRM.

MATERIALS AND METHODS

Chemical Composition

To study the chemical composition of MTA, we used the KVEX Delta 4460 X-ray Energy dispersive spectrometer, modified with Micro EDS software (BEMAX UK Ltd.), in conjunction with a Hitachi S520 scanning electron microscope. MTA was mixed with sterilized distilled water and allowed to set in a 37°C incubator with 5% carbon dioxide and moisture. The material was set on a glass cover slip that had been previously sterilized using alcohol and flaming. Five set specimens with different proportions of water and powder were examined. For quantitative X-ray analysis, the specimens were carbon-coated to a thickness of 100 nm and again mounted in the S520 using the quantum DVEX system. Acceler-
The setting times of test materials were determined according to the method recommended by the ISO (6). Before mixing, the test materials, mixing pads, spatulas, and flat glass plates were placed in a room with a temperature of 23 ± 1°C for 1 h. Each material was then mixed and placed in a circular metal mold (15 mm diameter and 5 mm high). The assembly, comprising mold and test material, was placed on a metal block in a cabinet at 37°C and relative humidity of 95 to 100%, 2 min after the start of mixing. After a further 30 s to 1 min, the indentor needle (Gillmore) was lowered vertically onto the surface of the material (1.0 mm/min) and allowed to remain there for 5 s. This process was repeated every 30 s until the needle failed to make a complete circular indentation in the test material. The setting time was the duration of time that elapsed from start of mixing to when the indentor needle failed to make an indentation in the material. This test was repeated six times for each material.

### Compressive Strength

The compressive strengths of test materials were determined according to the method recommended by the ISO (6). The instruments and the test materials were conditioned at 23 ± 1°C in the laboratory for 1 h before testing. Each material was mixed and placed in split stainless steel molds (12 mm high and 6 mm wide) within 2 min before start of mixing. Each mold was packed to excess and was placed on the bottom plate with the application of slight pressure. After removing any extruded material and placing the top plate, the mold and plates were clamped together. The whole assembly was transferred to an oven with a constant temperature of 37 ± 1°C for 3 h within 3 min after start of mixing. The specimens were removed from the molds and examined for voids and chipped edges. Defective specimens were discarded, and six acceptable samples were prepared for each test material for each time interval. The specimens were immersed in distilled water for 21 h or 3 wk before their compressive strengths were measured using an Instron 1185 Testing Machine (Instron Ltd., High Wycombe, UK). After placing each specimen with its flat ends between the platens of the apparatus and ensuring that the load was applied in the long axis of the test sample, the maximum load required to fracture each specimen was noted, and the compressive strength (C) was calculated in Megapascals using the formula

\[
C = \frac{4P}{\pi D^2}
\]

where \( P \) is the maximum applied load in Newtons, and \( D \) is the mean diameter of the specimen in millimeters (6).

One way analysis of variance with Duncan multiple comparisons were used to determine statistical differences between compressive strengths of the various test materials at both time intervals.

### Solubility

The degree of solubility of test materials was determined by a modified method of the American Dental Association (ADA) specification #30 (7). The materials were prepared in line with manufacturers’ recommendations. Super-EBA cement and MTA were hand-mixed, whereas amalgam and IRM were mixed in a machine. After mixing, each substance was made into a small disc

---

**Table 1. Material used for experiments**

<table>
<thead>
<tr>
<th>Material</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sybraloy amalgam</td>
<td>Kerr Mfg. Co., Romulus, MI</td>
</tr>
<tr>
<td>Super-EBA</td>
<td>Harry J. Bosworth Co., Skokie, IL</td>
</tr>
<tr>
<td>IRM</td>
<td>L. D. Caulk Co., Milford, DE</td>
</tr>
<tr>
<td>MTA</td>
<td>Loma Linda University, Loma Linda, CA</td>
</tr>
</tbody>
</table>

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The pH of MTA as it set was measured with a pH meter (Pye, Cambridge, UK) using a temperature-compensated electrode.

**Radiopacity**

The radiopacity of MTA was determined according to the method described by the International Organization for Standardization (ISO 6876), section 7.7 (6). After mixing, MTA was packed into a stainless steel ring mold with an internal diameter of 10 mm and a depth of 1 mm. The mold was placed on a glass slab before insertion of the material. It was covered with a glass slide and allowed to set for 3 h.

Radiographs were taken with a target-film distance of ~30 cm using a cardboard extension to the cone of the X-ray machine. The dental X-ray unit (Heliodent MD, Siemens, Bensheim, Germany) was set at 70 kV, a current of 7 mA, and an exposure time of 0.25 s to give a radiographic density reading of ~2 (including base and fog) for the exposed and processed film under the 1-mm thick section of the step wedge (Protex Ten-Step Aluminum Step wedge, Everything X-Ray Ltd., Watford, UK). The specimens were placed directly on the film packet. A total of five films were taken for each specimen. The films were processed in an automatic developing machine (P10, Hope Industries, Letchworth, UK).

The photographic densitometer (Melico/Photolog, B/W Transmission Densitometer, Model TDX, Medical and Electrical Instrumentation Ltd., London, UK) was used to take readings of the radiographic image of the specimens, each step of the step wedge, and the unexposed part of the film. Three readings were taken for each film and the mean calculated. The net radiographic density was derived from the following equation:

\[
\text{Net Radiographic Density} = (\text{Gross Radiographic Density}) - (\text{Base and Fog}).
\]

Graphs were plotted for log–net radiographic density of the aluminum steps versus thickness of aluminum (mm) for each group. These gave straight line plots, and from these the mean net radiographic density values of the materials were transformed into radiopacity expressed as equivalent thickness of aluminum.
EXAMINATION OF THE MTA SHOWS SPECIFIC PHASES THROUGHOUT THE MATERIAL. ALL MTA WAS DIVIDED INTO CALCIUM OXIDE AND CALCIUM PHOSPHATE. FURTHER ANALYSIS DEMONSTRATED THAT THE FORMER APPEARED AS DISCRETE CRYSTALS AND THE LATTER AS AN AMORPHOUS STRUCTURE WITH NO APPARENT CRYSTAL GROWTH BUT A GRANULAR APPEARANCE. THE MEAN VALUE OF THE PRISMS WAS 87% CALCIUM AND 2.47% SILICA, THE REMAINDER BEING OXYGEN. IN AREAS OF AMORPHOUS STRUCTURE, THERE SEemed TO BE 33% CALCIUM, 49% PHOSPHATE, 2% CARBON, 3% CHLORIDE, AND 6% SILICA.

THE CHANGE IN pH AS A FUNCTION OF TIME FOR MTA IS SHOWN IN FIG. 1. THE pH OF MTA AFTER MIXING WAS 10.2, AND IT ROSE TO 12.5 AT 3 h; THEREAFTER, IT REMAINED CONSTANT. THE MEAN RADIOACTIVITY FOR MTA WAS 7.17 mm OF EQUIVALENT THICKNESS OF ALUMINUM.

THE MEAN SETTING TIME AND STANDARD DEVIATION FOR THE TEST MATERIALS WERE AS FOLLOWS: AMALGAM, 4 min (±30 s); SUPER-EBA, 9 min (±30 s); IRM, 6 min (±30 s); AND MTA, 2 h 45 min (±5 min).

THE MEANS AND STANDARD DEVIATIONS OF COMPRESSIVE STRENGTH OF THE TEST MATERIALS AFTER THE TWO TIME INTERVALS ARE SHOWN IN TABLE 2. AMALGAM HAD THE HIGHEST COMPRESSIVE STRENGTH AMONG THE MATERIALS TESTED. STATISTICAL ANALYSIS OF THE DATA REGARDING THE REMAINING THREE MATERIALS SHOWED THAT THE COMPRESSIVE STRENGTH OF SUPER-EBA WAS SIGNIFICANTLY GREATER THAN THAT OF IRM AND MTA AFTER 24 h (p < 0.05); COMPRESSIVE STRENGTH OF IRM WAS SIGNIFICANTLY MORE THAN MTA (p < 0.05) AT THE SAME TIME INTERVAL. THE COMPRESSIVE STRENGTH OF ALL CEMENTS INCREASED AFTER 3 wk, AND THE STRENGTH OF SUPER-EBA WAS SIGNIFICANTLY HIGHER THAN THAT OF IRM (p < 0.05), BUT THERE WAS NO SIGNIFICANT DIFFERENCE BETWEEN THE STRENGTHS OF SUPER-EBA AND MTA AT THESE TIME INTERVALS.

THE MEAN WEIGHTS (g) OF SPECIMENS AND STANDARD DEVIATION FOR THE TEST MATERIALS AT VARIOUS TIME INTERVALS ARE SHOWN IN TABLE 3. STATISTICAL ANALYSIS OF THE DATA REGARDING WEIGHT LOSS CHANGES SHOWED NO SIGNIFICANT CHANGES FOR AMALGAM, SUPER-EBA, AND MTA WHEN THE MEAN WEIGHT OF THE SPECIMENS WERE COMPARED AT DIFFERENT TIME INTERVALS. HOWEVER, WHEN THE MEAN WEIGHTS OF IRM SPECIMENS WERE COMPARED BETWEEN DAY 1 AND THOSE ON DAYS 7 AND 21, SIGNIFICANT DIFFERENCES WERE NOTED (p < 0.001). SIMILAR DIFFERENCES WERE NOTED WHEN THE MEAN WEIGHT OF THE SPECIMENS WERE COMPARED AT DAY 7 Versus DAY 21.

DISCUSSION

Numerous investigations have been performed to study the physical properties of dental filling materials (8-13). However, information regarding the physical properties of root-end filling materials is scant. Owadally and Pitt Ford (14) evaluated the effect of adding 10% and 20% hydroxyapatite (HAP) on the physical properties of IRM and found that IRM had longer mean working and mean setting times than IRM + 10% or 20% HAP. IRM also had a greater compressive strength than both IRM + 10% HAP or IRM + 20% HAP. IN addition, their results indicated that adding 10% or 20% HAP to IRM causes disintegration of these materials in buffered phosphate and in bovine serum after 8 wk. IN contrast, IRM and EBA cements showed no noticeable signs of disintegration in the same media after 6 months.

MTA POWDER CONSISTS OF FINE HYDROPHILIC PARTICLES. THE PRINCIPLE COMPOUNDS PRESENT IN THIS MATERIAL ARE TRICALCICM SILICATE, TRICALCICM ALUMINATE, TRICALCICM OXIDE, AND SILICATE OXIDE. IN ADDITION, THERE ARE SMALL AMOUNTS OF A FEW OTHER MINERAL OXIDES THAT ARE RESPONSIBLE FOR THE CHEMICAL AND PHYSICAL PROPERTIES OF THIS AGGREGATE. BISMUTH OXIDE POWDER HAS BEEN ADDED TO MAKE THE AGGREGATE RADIOACTIVE. ELECTRON PROBE MICROANALYSIS OF MTA POWDER SHOWN THAT CALCIUM AND PHOSPHOROUS ARE THE MAIN IONS PRESENT IN THIS MATERIAL. BECAUSE THESE IONS ARE ALSO THE PRINCIPAL COMPONENTS OF DENTAL HARD TISSUES, MTA MAY PROVE TO BE BIOCOMPATIBLE WHEN USED IN CONTACT WITH CELLS AND TISSUES. IN VITRO AND IN VIVO STUDIES ARE IN PROGRESS TO TEST THE BIOCOMPATIBILITY OF THIS MATERIAL IN CELL CULTURE AND TISSUES. INDUCTION OF A HARD TISSUE BARRIER, SIMILAR TO THAT OBTAINED IN APEXIFICATION PROCEDURES, FOLLOWING ROOT-END FILLING MATERIAL 351

<table>
<thead>
<tr>
<th>Material</th>
<th>24 h</th>
<th>21 days</th>
</tr>
</thead>
<tbody>
<tr>
<td>Amalgam</td>
<td>312.5</td>
<td>311.1</td>
</tr>
<tr>
<td></td>
<td>(20.1)</td>
<td>(23.8)</td>
</tr>
<tr>
<td>Super-EBA</td>
<td>60.0</td>
<td>78.1</td>
</tr>
<tr>
<td></td>
<td>(5.5)</td>
<td>(9.3)</td>
</tr>
<tr>
<td>IRM</td>
<td>52.2</td>
<td>57.4</td>
</tr>
<tr>
<td></td>
<td>(3.4)</td>
<td>(5.8)</td>
</tr>
<tr>
<td>MTA</td>
<td>40.0</td>
<td>67.3</td>
</tr>
<tr>
<td></td>
<td>(4.4)</td>
<td>(6.6)</td>
</tr>
</tbody>
</table>
failing would minimize the interaction between material and host tissues. Because MTA has a high pH similar to calcium hydroxide cement, it is possible that induction of hard tissue formation may occur following the use of this substance as a root-end filling material.

Among other characteristics, an ideal root-end filling material should be more radiopaque than its surrounding structures when placed in a root-end cavity preparation. Shah et al. (15) determined the radiopacity of potential root-end filling materials using the same method utilized in our study and found that amalgam was the most radiopaque material tested (>10 mm equivalent thickness of aluminum). The radiopacity of other relevant test materials in their experiment was as follows: Kalzinol (7.97), gutta-percha (6.14), IRM (5.30), Super-EBA (5.16), and dentin (0.70). A comparison between the radiopacity of MTA and those of other potential root-end filling materials in Shah et al.'s (15) study shows that MTA is slightly less radiopaque than Kalzinol (reinforced zinc oxide-eugenol–based cement) and more radiopaque than IRM and Super-EBA. Because MTA is more radiopaque than conventional gutta-percha and dentin, it should be easily distinguishable on radiographs when used as a root-end filling material.

Hydration of MTA powder results in a colloidal gel that solidifies to a hard structure in less than 3 h. The characteristics of the aggregate depend on the size of the particles, powder-to-water ratio, temperature, presence of water, and entrapped air (2). In this study, the ISO method of determining setting time was used (6). The results showed that amalgam had the shortest setting time and MTA the longest. The setting time for IRM in this study was very similar to that found by Owadally and Pitt Ford (14). It has generally been considered that a potential root-end filling material should set as soon as it is placed in the root-end cavity without significant shrinkage. This condition would allow dimensional stability of the material after placement and less time for an unset material to be in contact with vital tissues. However, in general terms, the quicker a material sets the more it shrinks. This phenomenon may explain why MTA in previous experiments had significantly less dye (2, 3) and bacterial (4) leakage than other materials tested as root-end filling materials.

Compressive strength is an important factor to consider when a filling material is placed in a cavity that bears occlusal pressure. Because root-end filling materials do not bear direct pressure, the compressive strength of these materials is not as important as those materials used to repair defects in occlusal surfaces. In the present study, MTA initially had the lowest compressive strength among materials tested, but its value increased with time. In a previous report, the compressive strength of MTA was reported by mistake as being equal to amalgam (2). The increase in compressive strength of MTA required the presence of moisture. Longer term studies might provide further information on compressive strength of MTA in the presence of moisture. However, the compressive strength value obtained for MTA is similar to those obtained for Super-EBA, IRM, and zinc phosphate (69–117), (16).

Solubility is another factor in assessing the suitability of potential substances to be used as restorative materials in dentistry (8–13). Lack of solubility has also been stated as an ideal characteristic for root-end filling materials (1). Despite some advantages of controlled long-term clinical studies, variations in the oral environment among participating patients are a major drawback of such investigations. Because of the long setting time of MTA, the recommended methods by ISO (6) or ADA specification #30 (7) to test the solubility of MTA had to be modified.

From our results, it seems that amalgam, Super-EBA, and MTA showed no signs of solubility in water; despite statistical differences between the mean weights of IRM specimens at three time intervals, the clinical significance of the mean differences—0.0074 g (day 1 versus day 7) and 0.0037 g (day 7 versus day 21)—is questionable. Owadally and Pitt Ford (14) evaluated the rate of disintegration of some zinc oxide-eugenol cements utilizing a replica method in conjunction with scanning electron microscopy, and found that IRM and Super-EBA showed no noticeable signs of disintegration after 6 months in buffered phosphate solution.

Erosion of restorative filling materials can occur either by acids generated by bacteria, acids present in foods or beverages (17), or by mechanical wear. The root-end filling materials are normally in contact with periradicular tissue fluid until they are covered either with fibrous connective tissue or cementum. Clinically, biocompatible root-end filling materials with good sealing ability should generate little or no inflammatory response in periradicular tissues, and encourage formation of fibrous connective tissue and/or cementum covering the entire root-end. Mechanical wear, an important factor in coronal restoration, is not a significant factor in erosion of root-end filling materials.

Based on the results of this study, it seems that MTA has adequate physical properties for use as a root-end filling material.

We thank Mr. PMM Shah for determining the radiopacity of MTA and Dr. M. Sherriff for determining the pH of MTA.

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References


<table>
<thead>
<tr>
<th>Time</th>
<th>Amalgam</th>
<th>Super-EBA</th>
<th>IRM</th>
<th>MTA</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 day</td>
<td>5.3961 ± 0.1549</td>
<td>1.4484 ± 0.1147</td>
<td>1.1885 ± 0.0319</td>
<td>1.1794 ± 0.1100</td>
</tr>
<tr>
<td>7 days</td>
<td>5.3959 ± 0.1547</td>
<td>1.4466 ± 0.1148</td>
<td>1.1811 ± 0.0315</td>
<td>1.1814 ± 0.1122</td>
</tr>
<tr>
<td>21 days</td>
<td>5.3961 ± 0.1548</td>
<td>1.4465 ± 0.1144</td>
<td>1.1774 ± 0.0315</td>
<td>1.1746 ± 0.1130</td>
</tr>
</tbody>
</table>

A Word for the Wise

A recent article, written by a person not in the legal profession but who had once been, was attributed to a "recovering" lawyer.

I like that; will they form a Barristers Anonymous organization?

I. Solon