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Physical and mechanical properties of experimental dentinogenic pulp capping material

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DISSERTATION

PHYSICAL AND MECHANICAL PROPERTIES OF EXPERIMENTAL DENTINOGENIC PULP CAPPING MATERIAL

by

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ABSTRACT

Bioactive Inorganic Element (BIE) is a bioactive glass that has been developed and studied for bone regeneration, and dentinogenesis. **Objective**: To test the physical and mechanical properties (setting time, pH level, solubility, and compressive strength) of two groups of BIE-containing materials: 20% BIE (20% BIE, 60% Portland cement, and 20% bismuth oxide) and 40% BIE (40% BIE, 40% Portland cement, and 20% bismuth oxide), and compare them with Dycal® and Experimental Mineral Trioxide Aggregate (EMTA) as the control groups, with and without calcium chloride at three different concentrations (5%, 10%, and 15%). **Methods:** Ten samples in each group were tested for setting time, pH level, compressive strength, and solubility. Setting time was determined following the ISO specification 6876 using digital dial indicator. The pH level of the storage solutions of each specimen in deionized water was measured after 2 hours, and 1, 7, 21, and 28 days. The compressive strength was measured following the ISO specification 9917, and solubility was measured as a weight loss after storage in deionized water. The compressive strength and

solubility were measured at 1, 7, 21, and 28 days. Data were statistically analyzed with two-way ANOVA and Tukey's tests. **Results:** Dycal® showed the least setting time material, which was comparable to the 40% BIE + 10%, and 15% $CaCl_2$ ($p > 0.05$). EMTA showed the longest setting time ($p < 0.0001$). For the pH level, at 2 hours the lowest level was for the 40% BIE + 15% $CaCl_2$ group, and the highest was for EMTA group. During all time intervals, the lowest compressive strength was for the Dycal®, and the highest was for the 40% BIE ($p < 0.0001$). During all time intervals, the Dycal® was the most soluble material ($p <0.0001$), and the solubility levels of EMTA, 20% BIE, and 40 % BIE were comparable ($p > 0.05$). The pH level, compressive strength, and solubility of all tested material increased with time and decreased with $CaCl_2$ addition. **Conclusion:** The 40% BIE with 10% $CaCl_2$ showed promising physical and mechanical properties that could compete the Dycal® and EMTA when used in pulp capping.

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$2CaO.SiO_2$……	Dicalcium Silicate
$3CaO.Al_2O_3$……..	Tricalcium Aluminate
$3CaO.SiO_2$……	Tricalcium Silicate
$4CaO.Al_2O_3.Fe_2O_3$……	Tetracalcium Aluminoferrite
ALP.…….	Alkaline Phosphatase Enzyme
BIE.……...	Bioactive Inorganic Element
BMP…….	Bone Morphogenic Protein
$Ca(OH)_2$ …….	Calcium Hydroxide
$CaCl_2.2H_2O$…….	Calcium chloride dihydrate
CaO…….	Calcium Oxide
$CaSO_4.4H_2O$……	Gypsum
EMTA……..	Experimental Mineral Trioxide Aggregate
FeO……..	Ferrous Oxide
GI/RMGI…….	Glass Ionomer cement/ Resin Modified Glass Ionomer
GMTA…….	Gray Mineral Trioxide Aggregate
IRM……...	Intermediate Restorative Material of Polymer Reinforced Zinc Oxide-Eugenol (Reinforced with Poly-Methyl Methacrylate)
ISO……	International Organization for Standardization

K_2SO_4……	Potassium Sulfate
MgO……..	Magnesium Oxide
MTA…….	Mineral Trioxide Aggregate
Na_2O ……	Sodium Oxide
Na_2SO_4……..	Sodium Sulfate
OC……	Osteocalcin
P_2O_5……..	Phosphorus Pentoxide
SEM……..	Scanning Electron Microscope
SiO_2……..	Silica - Silicon Dioxide
Super EBA……	Reinforced Zinc oxide-Eugenol Cement (mixture of 32% Eugenol and 68% Ethoxy Benzoic Acid)
TBF-β1…….	Transforming Growth Factor Beta One
WMTA……..	White Mineral Trioxide Aggregate
ZOE……...	Zinc oxide/Eugenol

INTRODUCTION

1. INTRODUCTION

From the time dentistry became a discipline, dentists have dealt with caries excavation, aiming for preserving tooth structure and pulp vitality. The financial cost of pulp exposure is high and often requires either tooth extraction or root canal therapy. The procedure of pulp capping provides an alternative procedure to that is pulp capping. Pulp capping is a procedure where a material (liner or base) is placed directly over the exposed pulp (direct pulp cap), or over remaining caries (indirect pulp cap) in an attempt to induce the formation of reparative dentine, to maintain pulp vitality. This precludes the need for more extensive and expensive dental procedures (Bogen et al., 2008; Heys et al., 1981; Hilton, 2009).

By using the pulp capping technique over root canal treatment and saving the tooth vitality, the force of mastication defense mechanism is preserved. Endodontically treated teeth require 2.5 times more load to register a proprioceptive response than vital teeth (Stanley, 1989). In addition, pulp capping requires a less complex, and less expensive procedure than root canal treatment.

The requirements of an ideal material for pulp capping include such things as the ability to maintain the pulp vitality and function, the ability to form dentine bridge, adequate mechanical properties, adhesion to dentine, and the quality of being easy to handle (Stephen Cohen, 9th edition). Many different materials have been used in dentistry to form a good reparative dentin bridge and pulp healing; including Calcium Hydroxide, Zinc Oxide/Eugenol (ZOE), Glass Ionomer cement/ Resin Modified Glass Ionomer (GI/RMGI),

and Mineral Trioxide Aggregate (MTA). However, a Cochrane systematic review found there was no current reliable, nonabsorbable bioactive pulp capping material that induces cellular repair mechanisms to form a biologically stable dentin bridge, and seal the dentin (Bogen et al., 2008; Miyashita et al., 2007). Currently, the most commonly used pulp capping materials are calcium hydroxide and MTA.

Calcium hydroxide was considered the standard material for pulp capping that induces the formation of reparative dentin. However, long-term studies have shown that the material does not provide close adaptation to dentin, and it does not induce consistent odontoblast differentiation. Therefore, the resultant reparative dentin has the characteristic of tunnel defect. The presence of these channels in the dentin may facilitate the entry of microorganisms into the pulp tissue and cause more tooth infection (Aeinehchi et al, 2003; Cox et al, 1996; Kitasako et al, 2008).

MTA is a bioactive silicate cement, which has been shown to be an effective pulp-capping material. The success of MTA material in pulp capping is due to its dentin sealing ability, alkaline pH, and ability to induce pulpal cell proliferation. The main disadvantage of the material is the prolonged setting time, which in the presence of moisture may take up to 3 to 4 hours (Torabinejad et al., 1995(a)).

Since the invention of Portland cement in the mid-18th century, calcium chloride has been the most widely used cement accelerant. Studies have proven the biocompatibility and the osteoconductive properties of the accelerated Portland cement (Abdullah et al., 2002).

Furthermore, the setting time of MTA was successfully reduced by the addition of calcium chloride (AlAnezi et al., 2011).

Both calcium hydroxide and MTA materials do not exclusively stimulate dentinogenesis; instead, they induce the formation of mineralized tissue (hard tissue bridge) as a result of their high pH levels.

Bioactive Inorganic Element (BIE) is a biocompatible, bioactive glass material composed of silicon, calcium, and phosphorus. The material has been developed and studied for tissue engineering and bone regeneration at Boston University. Studies have also proven the dentinogenic effect of BIE material through stimulating pulp cell proliferation and differentiation (Al-Bazie, 2000; Ellis, 2003). However, the clinical use of this material as a pulp capping and dentin regeneration material has not been studied yet, due to the lack of laboratory experiments of its mechanical and physical properties, as well as the lack of in-vivo studies.

Despite the recognizable clinical applications of MTA and calcium hydroxide, both materials have some disadvantages. We are conducting this study with the aim of characterizing a novel pulp capping material with the dentinogenic effect because there is a gap in the literature in identifying the most reliable, nonabsorbable, bioactive pulp capping material that consistently stimulates cellular repair, seals the dentin, and promotes dentinogenesis. The objective of the study is to evaluate the physical and mechanical

properties of BIE containing materials, and compare it to calcium hydroxide and experimental mineral trioxide aggregate (EMTA) with and with out calcium chloride.

LITERATURE REVIEW

2. LITERATURE REVIEW

2.1. Calcium Hydroxide ($Ca(OH)_2$)

Nygren, introduced calcium hydroxide ($Ca(OH)_2$) to dentistry in 1838 for the treatment of the "fistula dentalis". In 1851, Codman conducted the first attempt to preserve dental pulp using calcium hydroxide. In 1936, calcium hydroxide became widely accepted in dentistry after Hermann's groundbreaking studies that demonstrated the effectiveness of calcium hydroxide in inducing the formation of secondary dentin over the vital pulp. In 1938, Teuscher and Zander introduced calcium hydroxide in the United States, and since that time reports of successful pulp healing have taken place in the literature. It is considered to be the best material to induces hard tissue formation, stimulate healing, and have a long-term record (up to 10 years) of clinical success as a direct pulp-capping agent (Fava & Saunders, 1999; Hilton, 2009).

Calcium hydroxide is a white, odorless powder with a molecular weight of 74.08. It is insoluble in alcohol, and it shows high solubility in water (0.4% - 78%) (Anusavica K, 11th edition). It is a strong base material with pH levels ranging from 9.2 to 12.8 (Fava & Saunders, 1999; John M. Powers, 2nd edition). Calcium hydroxide is considered to be a weak-strength material. The self-cure form has a low tensile strength (1 MPa), low compressive strength (12 - 26 MPa), and low modulus of elasticity (0.4 GPa) compared to other high-strength base materials. The low elastic modulus of the material restricts its usage to areas where mechanical support can be achieved by sound dentin or under a high-strength base material. If used in sufficient thickness, the calcium hydroxide material may provide some

thermal insulation to the pulp. However, it is not recommended to use thicknesses greater than 0.5 mm (John M. Powers, 2nd edition).

2.1.1. Forms and Compositions

Calcium hydroxide pulp capping materials are classified based on either their form, or their vehicle type.

1. Classification According to the Forms:

Calcium hydroxide is available in the market in different forms including the two paste system (common form), single paste, light cured system, and powered.

a. Two Paste System:

There are two pastes in the two paste system. The base paste contains calcium tungstate, disalicylate ester tribasic calcium phosphate, and zinc oxide in glycol salicylate. The catalyst paste contains calcium hydroxide, zinc oxide, and zinc stearate in ethylene toluene sulfonamide.

The calcium hydroxide and a disalicylate are responsible for the setting time. Calcium tungstate or barium sulfate is responsible for radiopacity (John M. Powers, 2nd edition). An example of calcium hydroxide contaning these materials is Dycal®, which is the most commonly used and best studied material so far.

b. Single Paste System:

An example of this system is a premixed calcium hydroxide methylcellulose (Pulpdent® Paste). Another example is a calcium hydroxide in a radiopaque hydroxyethylcellulose base (Multi-Cal, Pulpdent®).

c. Light Cured System (Prisma VLC Dycal®):

This system consists of calcium hydroxide and barium sulfate dispersed in a urethane dimethacrylate resin. This form contains camphorquinone (CQ) for initiation.

d. Powder Form:

This system consist of calcium hydroxide powder mixed with the different vehicle types.

2. Classification According to the Vehicle Type:

The vehicle used with calcium hydroxide represents an important component of the pastes, and it affects their physical, chemical, and clinical application. Listed below are three types of vehicles (Farhad & Mohammadi, 2005; Fava & Saunders, 1999; Mohammadi & Dummer, 2011).

a. Water-Soluble:

This type includes aqueous solutions such as water, saline, dental anesthetics, and Ringer's solution. In the market, such products as Calxyl®, and Pulpdent® Tempcanal are examples of calcium hydroxide in a water-soluble vehicle. This type of vehicle has the

highest degree of solubility, and therefore requires the patients to make multiple visits to the dentist for redressing the material.

b. Viscous Vehicles:

This type of vehicle includes such products as glycerine, polyethyleneglycol and propylene glycol. The product Calen™ is an example of the viscous form of calcium hydroxide. Viscous vehicles are also water-soluble but to a lesser extent. Thus, the application of the material requires the patient to make fewer visits to the dentist.

c. Oily Vehicles:

Include olive oil, silicone oil, camphor, and fatty acids. An example of the material in the market is the product Vitapex. This type of vehicle shows the lowest degree of solubility and the longest period of tissue contact with the material.

2.1.2. Mechanism of Action

In dentistry, calcium hydroxide plays an important role in mineralization and antimicrobial effects. The mineralization effect derives from the ability of calcium hydroxide to activate the alkaline phosphatase enzyme (tissue enzyme), producing calcium phosphate, which is the molecular unit of hydroxyapatite that participates in the formation of the hard tissue bridge.

Direct contact of calcium hydroxide with the pulp tissue, induces the formation of necrotic areas within a few minutes. The zone of necrosis is formed by altering the physical-

chemical status of intercellular substance through the rupture of glycoproteins. After that, the formation of mineralized tissue will be observed from about the seventh to the tenth day (Estrela & Holland, 2003).

The antimicrobial effect also derives from the high pH of calcium hydroxide and from the release of hydroxyl ions, which are highly oxidant free radicals. Both of these qualities are responsible for bacterial death because they alter the integrity of the cytoplasmic membrane of bacterial cells, cause damage to bacteria's DNA, and create denaturation of bacterial proteins (Estrela & Holland, 2003; Fava & Saunders, 1999).

2.1.3. Biological and Antimicrobial Properties

Biocompatibility:

The biocompatibility of the material comes from the following different mechanisms (Hilton, 2009):

a. The high pH level of calcium hydroxide causes necrosis in superficial layers of the pulp and mild irritation to the pulp. This will initiate an inflammatory response, and in the absence of bacteria it heals with a hard tissue barrier.

b. Calcium hydroxide can activate tissue enzymes such as alkaline phosphatase, which has an optimum pH level of 8.6 to 10.3. This enzyme induces the release of phosphate ions

that react with calcium ions to form calcium phosphate (the molecular unit of hydroxyapatite).

c. Calcium hydroxide solubilizes Bone Morphogenic Protein (BMP) and Transforming Growth Factor-Beta One (TBF-β1) from the dentin. These proteins are mediators for pulp repair.

Antimicrobial effect:

The high pH of calcium hydroxide causes chemical injury to the organic components and destruction of phospholipids or unsaturated fatty acids of the cytoplasmic membrane of the microorganism, which alter its integrity (Estrela & Holland, 2003).

2.1.4. Setting Time

According to the manufacturer of Dycal® DENTSPLY, the material takes 2.5 to 3.5 minutes for setting (at 23°C with 50% relative humidity). Setting time will reduce in the presence of increased humidity and/or temperature. Thus, the material will set faster inside patient mouth.

Several studies have examined the setting time of calcium hydroxide (Dycal®) and used it as the control group in comparison to other materials. The setting time of this material ranged from 60 to 145 seconds (Louwaku & Lertchirakarn, 2012; Qingyi Shen et al., 2010).

2.1.5. pH

The release of hydroxyl ions from calcium hydroxide cements generates an alkaline pH environment, that plays a critical role in dentin remineralization and protects against microorganism survival. Calcium hydroxide is a strong base material with pH levels ranging from 9.2 to 12.8 (Fava & Saunders, 1999; John M. Powers, 2nd edition; Tam, 1989).

In 2004, Pacios et al., tested the pH level of calcium hydroxide over 1, 7, 14, and 21 days solubilized in different vehicles: distilled water, camphorated monochlorophenol (CMCP), normal saline, cresatin, glycerin and propylene glycol (PG). The results showed no significant difference in pH levels for the different time intervals. However, the pH level of calcium hydroxide mixed with distilled water was significantly higher than those mixed with anesthetic or chlorhexidine solutions (Pacios et al, 2004).

In 2012, Louwaku and Lertchirakarn found that the pH level of calcium hydroxide (Dycal®) increased over time. It increased from 9.80 at 1 hour to 10.57 at 7 days and remained constant afterward for 7 days.

2.1.6. Compressive Strength

Calcium hydroxide is generally a weak material that can be used as a lining material for pulpal recovery, provide insulation under metallic restorative materials, and act as a chemical barrier under composite resins. The material shouldn't be used in thickness greater than 0.5 mm (John M. Powers, 2nd edition).

In 1989, Tam et al., compared the compressive strength of different forms of dental cement material including calcium hydroxide, calcium aluminate cement, and glass-ionomer cements at time intervals of 1 hour, 24 hours, 7 days, and 28 days. The results showed that calcium hydroxide cement had the lowest compressive strength among the materials, ranging from 6.2 ± 1.8 MPa to 48.0 ± 15.6 MPa at 1 hour and its strength increased over time to a range of 6.2 ± 0.5 MPa to 61.4 ± 19.4 MPa at 1 month.

Different studies tested the compressive strength of the material. A wide range of compressive strengths was recorded from 7.8 MPa to 30 MPa at 24 hours (Milosevic, 1991). The compressive strength of Dycal® at 24 hours was 17.09 ± 2.91 MPa (Louwaku & Lertchirakarn, 2012). Another study showed a decrease in the compressive strength of the material from 18.2 ± 2.8 MPa at 48 hours to 16.5 ± 4.7 MPa at 7 days; however, this decline was not statistically significant (Natale et al., 2015).

2.1.7. Solubility

Some solubility of the cement material is beneficial for the release of hydroxyl ions. At the same time, it causes material dissolution and leaves empty space beneath a restoration, resulting in tissue necrosis. Calcium hydroxide is a soluble material and should not be place in restoration margins. Prisma VLC Dycal exhibited the least acid solubility (0.02%) after 60 seconds immersion in 37% solution of phosphoric acid, when compared to other forms of calcium hydroxide, calcium aluminate cement, and glass-ionomer cements (Tam et al, 1989). Another study reported a 3.15 ± 0.22% disintegration of Dycal® at 24 hours (Louwaku & Lertchirakarn, 2012).

2.1.8. Summary of the Advantages and Disadvantages of Calcium Hydroxide

The advantages of pulp capping material containing calcium hydroxide are many. It has a long track record of success in vital pulp therapy (pulp capping, pulpotomies, root amputation, apexification, and apexogenesis), and it has low thermal conductivity, is easy to manipulate, inexpensive, has a short setting time, is biocompatible and releases hydroxide and calcium ions upon dissolution. The hydroxide ions raise the pH to approximately 12, and this high alkaline level creates antimicrobial and anti-inflammatory effects. Calcium ions boost the activity of the alkaline phosphatase enzyme, which aids in the maintenance of dentin mineralization and the formation of a dentin bridge (Hilton, 2009; O'Brien, 3rd edition; Qingyi Shen et al, 2010; Roberts et al, 2008).

On the other hand, pulp capping material containing calcium hydroxide has several disadvantages, including low strength (compressive, tensile), low modulus of elasticity, solubility that increases with time and with phosphoric acid or ether (precaution should be taken after acid etching), high cytotoxicity (from its high pH level), poor sealing ability (no inherent adhesive properties), and the induction of tunnel defect in the reparative dentine (Mickenautsch et al, 2010; O'Brien, 3rd edition). In addition, this material does not exclusively stimulate dentinogenesis; instead, it induces the formation of mineralized tissue as a hard tissue bridge (Gupta, 2011; Hilton 2009).

2.2. Mineral Trioxide Aggregate (MTA)

In the 1990s, Mahmoud Torabinejad developed Mineral Trioxide Aggregate (MTA) as a root end fillings material at the Loma Linda University. In 1993, Torabinejad first described the material in the dental literature (Lee & Torabinejad, 1993). In 1998, the U.S. Food and Drug Administration approved the material for endodontic use (Schmitt & Bogen, 2001).

The clinical application of MTA has been extended to include direct and indirect pulp capping, pulpotomies, treatment of external and internal resorption, and treatment of teeth with incomplete root formation. In the United States, MTA is manufactured by DENTSPLY/Tulsa Dental (Tulsa, OK, USA) under the trade name ProRoot® MTA.

2.2.1 Composition

MTA powder consists of fine, hydrophilic particles that are composed of 75% Portland cement, 20% bismuth oxide, and 5% gypsum by weight and contain trace amounts of SiO_2, CaO, MgO, K_2SO_4, and Na_2SO_4. The main component, Portland cement, is a mixture of dicalcium silicate ($2CaO.SiO_2$), tricalcium silicate ($3CaO.SiO_2$), tricalcium aluminate ($3CaO.Al_2O_3$), gypsum ($CaSO_4.4H_2O$), and tetracalcium aluminoferrite ($4CaO.Al_2O_3.Fe_2O_3$) (Camilleri et al., 2005 (a); Dammaschke et al., 2005; Sarkar et al., 2005). Gypsum is present in both Portland cement, and in MTA to retard the setting reaction. Bismuth oxide is added to MTA for radiopacity (Camilleri, 2005(b); Roberts et al., 2008).

Compared to Portland cement, MTA has a smaller and more uniform particle size, contains fewer heavy toxic metals (e.g. manganese and strontium), has a lower concentration of gypsum, and has a longer working time (Abdullah et al., 2002; Islam et al., 2006). There are two types of MTA: gray MTA (GMTA), and white MTA (WMTA). White MTA was introduced in 2002 as ProRoot MTA to address esthetic concerns. Asgary et al. conducted a study in 2005 to look at the differences between the two materials using scanning electron microscopy (SEM) and electron probe microanalysis. The results showed that the main differences were: WMTA had lower concentrations of Al_2O_3 (54.9% less), MgO (56.5% less), and FeO (90.8% less). WMTA also had smaller particle sizes than GMTA. The lighter color of WMTA was attributed to the reduction of iron and magnesium (Asgary et al., 2005; Bortoluzzi et al., 2006; Dammaschke et al., 2005; Roberts et al., 2008).

2.2.2. Biological and Antimicrobial Properties

Calcium hydroxide is the primary reaction product of MTA with water. Thus, the biocompatibility and the antimicrobial properties that the material inherits are actually driven from the effects of calcium hydroxide (Camilleri, 2008; Camilleri & Ford, 2006; Hilton, 2009; Roberts et al., 2008). Increased calcium concentration is important for improving cell attachment efficiency and proliferation rate. The high pH creates an antibacterial environment, modulates cytokine production, promotes differentiation and migration of hard tissue–producing cells, and forms HA (or carbonated apatite) on the MTA surface, which provides a biological seal (Malhotra & Mala, 2013; Sarkar et al., 2005).

Different studies in the literature have documented the biocompatibility of MTA material both in vitro (Camilleri et al., 2004; Mitchell, 1996; Mitchell et al., 1999) and in vivo (Holland et al., 2001; Holland et al., 1999; Torabinejad et al., 1995(b); Queiroz et al., 2005). The material showed significantly less inflammatory response than Super EBA when both materials were implanted into the guinea pig (Torabinejad et al., 1995(b)). In 2000, Zhu et al. showed attachment and growth of osteoblasts on MTA, forming a monolayer. Osteoblasts had a more favorable response (good attachment and spread over the material) to MTA compared to IRM, composite resin and Amalgam (Zhu et al., 2000).

2.2.3. Setting Time

According to the manufacturer, MTA powder is mixed with sterile water in a ratio of 3:1 to form a colloidal gel, which takes approximately 3 to 4 hours, to solidify and form a hard structure (Torabinejad, 1995 (a)). The setting reaction includes the interaction of tricalcium silicate and dicalcium silicate with water to produce hydrated salt and calcium hydroxide (Dammaschke et al., 2005). The mean setting times of GMTA is longer (296.2 ± 18.9 minutes) WMTA (275.7 ± 11.4 minutes) (Chng et al., 2005; Islam & Yap, 2006). The setting time of MTA was measured in multiple studies: 133.10 ± 7.84

The long setting time of MTA is one of the main drawbacks to using the material in dental practice. Multiple studies have been conducted to discover ways to overcome this problem by adding different accelerators including calcium chloride ($CaCl_2$), calcium nitrite/nitrate (CN/N), and calcium formate (CF). The most commonly used accelerator is calcium chloride.

2.2.4. pH

Several studies have shown that MTA is highly alkaline. It has an initial pH of 10.2 when mixed with water, and this alkalinity level increases to 12.5 three hours after mixing and remains constant after that (Camilleri et al., 2005 (a); Felippe et al., 2006; Fridland & Rosado, 2005; Lee et al., 2011; Prasad et al., 2015; Torabinejad et al., 1995 (a)). GMTA exhibited a significantly lower pH level than WMTA and Portland cement at 60 minutes (Islam & Yap, 2006). The high pH value of MTA could be attributed to the constant release of calcium from MTA and the formation of calcium hydroxide.

2.2.5. Compressive Strength

MTA has significantly lower compressive strength (40.0 ± 4.4 MPa) than that of amalgam (312.5 ± 20.1 MPa), IRM (52.2 ± 3.4 MPa), and Super-EBA (60.0 ± 5.5 MPa) after 24 hours. However, at 3 weeks, there was no significant difference observed between the compressive strength of MTA, IRM, and Super-EBA (67.3 ± 6.6 MPa, 57.4 ± 5.9 MPa, and 78.1 ± 9.3 MPa, respectively), and the amalgam material retained the highest strength among the group (311.1 ± 23.8 MPa) (Torabinejad, 1995(a)). Dicalcium silicate was reported to be responsible for MTA strength (Dammaschke et al., 2005). The compressive strength, push-out strength, and retention strength of the material increased with time (up to 21 days) and in the presence of moisture, due to the prolonged maturation process of MTA (Malhotra & Mala, 2013). Jeong et al, (2010) demonstrated the same finding of increased compressive strength over time. The compressive strength of MTA increased from 27.37 ± 2.99 MPa to 59.89 ± 3.05 MPa at 24 hours and 7 days, respectively. Natale et al., (2015) documented a

lesser degree of increase in strength over time, ranging from 16.1 ± 5.0 MPa at 48 hours to 18.0 ±6.5 MPa and 7 days.

Multiple factors may influence the compressive strength of MTA. One of these factors is the type of MTA used. A significantly higher compressive strength was found with GMTA than with WMTA (Islam & Yap, 2006). Another factor is the mixing solution; powder mixed with sterile water showed higher compressive strength compared to powder mixed with chlorhexidine (Holt et al., 2007). In addition, the presence of moisture was found to increase the compressive strength of the MTA material (Torabinejad et al., 1995 (a))

On the other hand, a lower compressive strength was documented when the material was etched by 37% phosphoric acid. Thus, the authors of that study recommended a waiting time of at least 96 hours for placing a restoration that requires acid-etching following MTA placement (Kayahan et al., 2009).

2.2.6. Solubility

Several short-term studies reported minimal or no solubility of MTA when placed in distilled water (Fridland & Rosado, 2005; Islam et al., 2006; Torabinejad et al., 1995 (a)). However, most long-term studies reported an increase in solubility over time, and few studies revealed a 24% material loss after 78 days of storage in water (Fridland & Rosado, 2003; 2005; Hilton, 2009). The low solubility of MTA is attributable to bismuth oxide addition, which is insoluble in water and reacts with both calcium and silicate (Camilleri, 2007; Parirokh, 2010; Rao & Shenoy, 2009).

A 2003 study found that increasing the water-to-powder ratios of MTA significantly increased the solubility of the material (Fridland & Rosado, 2003). In addition, WMTA displayed significantly more solubility than GMTA (Islam & Yap, 2006). Gandolfi et al. (2013) found that the solubility of MTA was 10.70 ± 0.33% after 24 hours of immersion in deionized water.

2.2.7. Summary of the Advantages and Disadvantages of MTA

The material has shown advantages over calcium hydroxide in its superior sealing and marginal adaptation, as well as its potential to be used in the presence of moisture. MTA was developed and recommended for many applications such as root-end filling, perforation repair, apexification, vital pulp therapy, and apical barrier formation for teeth with necrotic pulps and open apexes (Parirokh, 2010; Parirokh et al., 2011).

Although the material has shown some advantages for use in pulp capping, there are several disadvantages to using MTA, such as the prolonged setting time, the difficulty of handling the material, and tooth discoloration (with GMTA) (Torabinejad et al., 1995 (c)). The long setting time requires that the process of pulp capping to be done either in a two-step procedure or with the use of a quick-setting liner to protect the MTA during permanent restoration placement (Hilton, 2009). Abdullah et al., (2002) found that a reduction in the setting time of Portland cement (the major component of MTA) could be achieved through the addition of calcium chloride. Another limitation of MTA is the high solubility of the material. It demonstrated 24% loss after 78 days of storage in water (Fridland & Rosado, 2003, 2005; Hilton, 2009).

There are several reports concerning the price of MTA, which may limit the widespread use of the material (Casas et al., 2005; Srinivasan & Waterhouse, 2006). The cost of a single use of MTA is approximately $60 - $75 USD, and a single gram of MTA powder costs approximately the same as 24 grams of calcium hydroxide (Srinivasan & Whitworth, 2009).

2.3. Calcium Chloride ($CaCl_2$)

Calcium chloride ($CaCl_2$) is a colorless, crystalline solid basic chemical material, available in both hydrous and anhydrous form. The U.S. Food and Drug Administration lists the material as "Generally Recognizable As Safe" (GRAS). Therefore, it is used in many food products (Food Ingredients and Colors, 2004)

Since its first use in 1885, the material has been widely used as an accelerator for concrete (Mailvaganam & Rixom, 1999). It has a wide application in the medical field, including for the treatment of hypocalcaemia, cardiac resuscitation, and open heart surgery to improve myocardial contraction. It is also used in a variety of bone grafting techniques for the coagulation process (Marx et al., 1998).

Calcium chloride does not interfere with the biocompatibility of Portland cement, the main component of MTA (Abdullah et al., 2002). Several in vitro and a few in vivo studies have shown that the addition of calcium chloride to MTA improved its handling characteristics and calcium ion release, and lowered its pH level without affecting its

biocompatibility. The results of a preliminary investigation on dogs' teeth showed promising results regarding the use of MTA/$CaCl_2$ as a pulpotomy agent (Parirokh et al., 2011).

In 2011, Parirokh et al. compared histological changes in canine dental pulp treated either with MTA or MTA containing 10% $CaCl_2$ as pulp capping agent. The results showed no statistically significant difference in the response of teeth to both materials as pulp-capping agents ($p > 0.05$). However, higher numbers of chronic inflammatory cells and necrosis as well as incomplete calcified bridge formation were seen in teeth capped with MTA + 10% $CaCl_2$ compared to teeth capped with MTA alone.

Calcium chloride has been found to decrease the setting time of MTA and improve the sealing ability of MTA (Bortoluzzi et al., 2006; Hilton, 2009). In 2007, Wiltbank added three different Portland cement (PC) accelerators (calcium chloride, calcium formate, and calcium nitrate) to GMTA, WMTA, and PC. Initial setting time, dimensional stability, and pH were measured. The results showed that only calcium chloride significantly reduced the setting and pH level of all tested materials. Calcium formate also decreased the setting time of all tested materials; however, it increased the material pH level causing tissue necrosis. Calcium nitrate reduced the setting time of only two tested materials (GMTA and PC), and it increased the pH level of all tested materials. Dimensional stability was not significantly different between control and experimental groups in this study.

The resultant rate of heat production and intensity has been found to increase following the chemical reaction of adding calcium chloride to MTA. Thus, the compressive,

tensile, and flexural strength of the material increased temporarily at the early stage, and then declined. Adding 10% $CaCl_2$ improved the physical properties of MTA and Portland cement, it reduced their setting times and solubility, and decreased the pH level of the cements (Prasad et. al., 2015). The fact that $CaCl_2$ penetrates into the pores of the cement might provide an explanation for the acceleration of the setting time of the material. That has been shown to leads to acceleration of the hydration process of the silicates, and facilitation of the crystallization processes of the material (Abbaszadeganet et al., 2015; Kogan et al., 2006).

2.4. Bioactive Glasses

Bioactive glasses are groups of surface-active osteoconductive and osteoinductive materials capable of forming a chemical bond with bone when used as a bone graft substitute. The materials were first discovered in the 1960s by Larry Hench at the University of Florida, and they show an excellent degree of biocompatibility with hard and soft tissue structure with no or little inflammatory responses in vivo studies (Wilson et al., 1981).

Bioactive glasses are mainly composed of SiO_2, CaO, Na_2O and P_2O_5. The properties of the materials depend mainly upon the different proportions of the above-mentioned oxides. Thus, different brands and types of the bioactive glasses are available. The materials work when a silica-based carbonated hydroxyapatite layer is formed after the chemical bond of the bioactive glass to bone (Hench & Wilson, 1984).

The materials have been used for a long time in both the medical and the dental field. They have been used in orthopedics and surgical procedures such as bone reconstruction

(especially iliac crest and facial bone defects). Bioactive glasses are also used for treatment of periodontal bone defects and sinus lifts (Asano et al., 1994; Wilson et al., 1981).

2.5. Bioactive Inorganic Element (BIE)

A bioactive inorganic element (BIE) is a type of bioactive glass that has been developed and studied for biocompatibility in tissue engineering, bone regeneration, and odontogenic properties at Boston University. It is composed of silicon, calcium and phosphorous in ratio of 6:2:1, and is formed through a sol-gel process (Al-Bazie, 2000; Ellis, 2003). The phosphorous and calcium elements in BIE are osteogenic and thus induce bone formation. The material has been examined as a framework support for tissue engineering and bone regeneration (Tang, 2006).

Adding BIE to MTA has been shown to increase the expression of alkaline phosphatase (ALP) enzyme and osteocalcin (OC) in human osteoblasts and odontoblasts (Al-Bazie, 2000). Alkaline phosphatase enzymes are present in high amounts during dentinogenesis, and osteogenesis. Osteocalcin is a small protein produced during matrix mineralization. High expression of osteocalcin has been found to indicate mineralized tissue forming cells (Yokose et al., 2000). Thus, this enzyme is an indicator of osteogenesis.

Dentin Sialoprotein (DSP), and Dentin Phosphoprotein (DPP) are considered markers of dentinogenesis (Butler & Ritchie, 1995; Ruch et al., 1995), and they are produced only by odontoblasts. DSP is present only in young and mature odontoblasts, dentine, and pre-ameloblasts (Bronckers et al., 1993; D' Souza et al., 1992) . Although the function of DSP is

unknown, its appearance in both odontoblasts and pre-ameloblasts may suggest a role in epithelial mesenchymal signaling, which takes place during the cellular development of teeth (Butler et al., 1992). Thus, it has to be considered a hallmark of odontoblast phenotype and/or existence of dentin.

At Boston University, Ellis (2003) conducted a study that assessed the effects of BIE on human dental pulp. He found that DSP was elevated in the presence of BIE, which is indicative of dentinogenesis. The biocompatibility of BIE material has been previously studied and has yielded good results, however, its mechanical properties have not been investigated (Al-Bazie, 2000).

2.6. Effect of Particle Size on Material Characteristics

Taking into account morphological characterization, the particle size of cement has been found to be an important factor in the characterization feature of its physical properties (Dammarchke et al., 2005). Several studies have investigated the relationship between cement particle size, hydration kinetics, and the strength properties of the material. It has been shown that the small particle size increases the surface area available for hydration and improves early properties such as higher early strengths (Bentz et al., 1999; Frigioine, 1976; Osbaeck, 1989).

Particle size of the cement has also been found to affect the handling characteristics of these materials. Smaller particles enhance surface contact with the mixing liquid and result in

a greater strength as well as ease of handling.

Calcium hydroxide material and MTA are both used in pulp capping. However, both materials have several limitations. These limitations include the tunnel defect in reparative dentin (which allows bacterial penetration into the pulp) and the prolonged setting time of MTA. Additionally, neither calcium hydroxide nor MTA can induce dentinogenesis; instead, they induce the formation of necrotic mineralized tissue.

Since the biocompatibility of BIE and its ability to induce dentinogenesis have been studied in vitro before at Boston University, the aim of this study is to test the physical and mechanical properties (setting time, pH level, solubility, and compressive strength) of BIE-containing materials and compare it to calcium hydroxide (Dycal®) and Experimental MTA (EMTA) with and without calcium chloride for use as a pulp capping material.

HYPOTHESIS

3. HYPOTHESIS

Null Hypothesis: there is no difference in the physical and mechanical properties of BIE- containing materials compared to Dycal® and EMTA, with or without calcium chloride.

Alternative Hypothesis: There is a difference in the physical and mechanical properties of BIE- containing materials compared to calcium hydroxide and EMTA, with or without calcium chloride.

OBJECTIVES OF THE STUDY

4. OBJECTIVES OF THE STUDY

Objective-1:

This study will determine the setting time of various concentrations of BIE-containing material and compare it to Dycal® and EMTA with and with out calcium chloride.

Objective-2:

This study will determine the pH level of various concentrations of BIE- containing material and compare it to Dycal® and EMTA with and with out calcium chloride.

Objective-3:

This study will determine the compressive strength of various concentrations of BIE-containing material and compare it to Dycal® and EMTA with and with out calcium chloride.

Objective-4:

This study will determine the solubility of various concentrations of BIE- containing material, and compare it to Dycal® and EMTA with and with out calcium chloride.

MATERIALS AND METHODS

5. MATERIALS AND METHODS

5.1. Materials:

Table 5-1: List of Materials:

Material	Manufacturer	Lot Number	Location
Type I Portland Cement	Lafarge	N/A	Herndon, VA
Bismuth Oxide	Ferro Electric	120321	Penn Yan, NY
Gypsum	US Gypsum Co.	198939	Chicago, IL
Calcium Chloride Dihydrate	Fisher Scientific	037734	Fair Lawn, NJ
Bioactive Inorganic Element	Courtesy of Dr. Chou	N/A	Boston University
Dycal®	DENTSPLY Caulk	023552	Milford, DE

Table 5-2: List of Equipment:

Item	Manufacturer	Model Number	Location	Description
Triple Purpose Timer	VWR	62344-904	Batavia, IL	Stopwatch
Gyrotory Shaker	New Brunswick Scientific CO., Inc.	G2	Edison, NJ	Shaker
Accumet pH Meter	Fisher Scientific	S68166	Springfield, NJ	pH Meter
Digital Caliper	SPI	N/A	Garden Grove, CA	Caliper
Instron Universal Testing Machine	Hitachi	SU6600	China	Universal Testing Machine
Scanning Electron Microscope	Philips	XL-20	Eindhoven, Netherlands	Scanning Electron Microscope
Sieve	VWR	#635	West Chester, PA	Sieve
Mitutoyo	MTI Corp.	F-150E	Aurora, IL	Digital Dial Indicator Microgauge
Spatula	Health	324	N/A	Spatula
Glass slab	Buffalo Dental	78570	Syosset, NY	Glass Slab
Pipette	Fisher Brand	1367811D	Pittsburgh, PA	Pipette
Rotating Electric Motor	Arrow Engineering CO., Inc.	850	Hillside, NJ	Mixing Powder
Finnpipette (4-200 μL)	Labsystem	18669640039	N/A	Pipette
ProPpette	MTC.Bio	P6080	Metuchen, NJ	Pipette Controller

5.2. Methods

5.2.1. Specimen Preparation

5.2.1.1. Preparation of Experimental Mineral Trioxide Aggregate (EMTA)

Based on the composition of ProRoot® MTA provided by the manufacturer, an experimental version of MTA (EMTA) was formulated similar to ProRoot® MTA. By mixing the following ingredients, four batches of 200 g EMTA were prepared:

150.0 g Type 1 Portland cement (LaFarge NA, Herndon, VA)
40.0 g Bismuth oxide (Lot # 120321)
10.0 g Gypsum Terra Alba (Lot # 198939, US Gypsum Co, Chicago, IL)

The ingredients were placed in 1- liter Nalgene® bottles, which were strapped to a slowly rotating electric motor for 24 hours to obtain a uniform and well-mixed powder via a tumbling motion (Figure 5-1).

Figure 5-1: Powder mixing using a slow rotating electric motor for 24 hours.

5.2.1.2. Preparation of Bioactive Inorganic Element (BIE) Powered

To reduce the particle size of the BIE material, 1- liter Nalgene® bottles filled with BIE powder together with zirconia balls at a measurement of 12.7 × 12.7mm (Cole-Parmer) were placed in a milling machine for 48 hours (Figure 5-2). Then the powder was sifted using # 635 sieve (VWR International, West Chester, PA) to collect particles smaller than 10 microns.

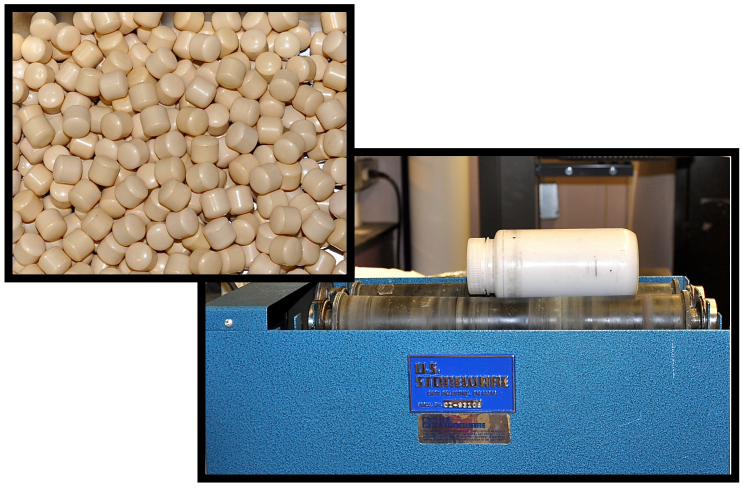


Figure 5-2: BIE powder with zirconia beads in a slow rotating milling machine for 48 hours.

5.2.1.3. Preparation of Bioactive Inorganic Element (BIE) Aggregates

The study aimed to establish two new materials that are similar to MTA but have better odontogenic properties. As mentioned previously, MTA is mainly composed of Type 1 Portland cement (75%), bismuth oxide for radiopacity (20%), and gypsum (5%). To establish new materials that are similar to MTA, the percentage of the radiopacifier (bismuth oxide) was standardized to 20%, the concentration of Portland cement was reduced in two different concentrations, and two different concentrations of BIE material were added (Table 5-3).

Four batches of 200 g of each group were prepared by mixing the ingredients listed in Table 5-3. Ingredients of each group were then placed in 1- liter Nalgene® bottles, which were strapped to a slowly rotating electric motor for 24 hours to obtain a uniform and well-mixed powder via a tumbling motion.

Table 5-3: BIE Groups and Compositions.

BIE group name	Portland Cement Weight g (%)	BIE Weight g (%)	Bismuth Oxide Weight g (%)
20% BIE	160 (60%)	40 (20%)	40 (20%)
40% BIE	80 (40%)	80 (40%)	40 (20%)

5.2.1.4. Preparation of Test Samples

A constant methodology was followed throughout the study for sample preparation. Except for Dycal®, all samples were prepared by hand mixing the powders with deionized water at room temperature (37° C) on a glass petri dish. Between experiments and before mixing, all armamentaria (molds, spatula, and petri dish) were cleaned and rinsed with deionized water and stored in a 37° C oven for a period of an hour. Samples were mixed at water to powder ratio of 3:1 (30% water) using 10 ml pipets for precise measurements. The amount of water in calcium chloride dihydrate ($CaCl_2.2H_2O$) was calculated and subtracted from the total weight to keep a constant ratio of water to powder. Three different concentrations of $CaCl_2$ (5%, 10%, and 15%) were added to the EMTA, the 20% BIE and the 40% BIE materials. Dycal® was prepared following the manual instructions from DENTSPLY. It arrived from the manufacturer in the form of two pastes (base and catalyst), and it was prepared by hand mixing equal amount of the base and catalyst on the provided mixing pad.

Mixing time was standardized for all samples to 60 minutes using a spatula, and packing each sample was done in 10 seconds. To achieve a flat surface, the surfaces of the samples were planed flush with the surface of the mold using a single-edge razor blade (VWR International, West Chester, PA).

5.2.2. Comparison of Particle Size Using SEM

Since the particle size of the material plays an important role in determining its physical properties, the particle sizes of all tested materials were evaluated with the scanning electron microscope (SEM). Individual samples were prepared for SEM analysis, and samples were visually examined and measured under the SEM with the goal of finding variations in particle size.

For sample preparation before using the SEM, the materials' powder was dispersed on an aluminum stub covered with a double-sided graphite sticker and sputters coated with gold/palladium under vacuum (Technics Hummer V sputter coater, USA) at 10 mA for 60 seconds. After that, samples were examined under the SEM (Hitachi SU6600, China) at 15.0kV and a working distance of 10 mm from the electron emission beam source. Samples were examined at magnification of 5.00k, using imaging software (QUARTS PCI Imaging, Vancouver BC).

To ensure that all materials were within the same particle size range, materials with larger particle sizes compared to the Dycal® were subjected to further grinding and were then sieved.

5.2.3 Setting Time

A modified method of International Organization for Standardization (ISO) 6876 (specification for dental root canal sealing materials) was used for testing the setting time. A Delrin ® mold containing three identical wells was used to achieve standardized samples dimensions (Figure 5-3). Each well had an inner diameter of 12.7 mm and a thickness of 4 mm. Mixing and packing the samples was conducted as described before in preparation of test samples.

The setting time test was performed using a Mitutoyo F-150E digital dial indicator (Mitutoyo MTI Corp. Aurora, IL) with a tip diameter of 1.5 mm and a measuring force of 120 g (Figure 5-4). After the sample was packed, the dial indicator tip was raised and placed on the mold well edge adjacent to the sample surface, where the digital reading was set to zero. The tip was elevated and carefully lowered onto the specimen surface at the appropriate time interval (Appendix Tables 10-28 through 10-40). The reading of the tip penetration was recorded after 10 seconds of contacting the tip to the specimen. Setting time was defined when the tip penetration reached 0.01 mm in 10 seconds in three different trials.

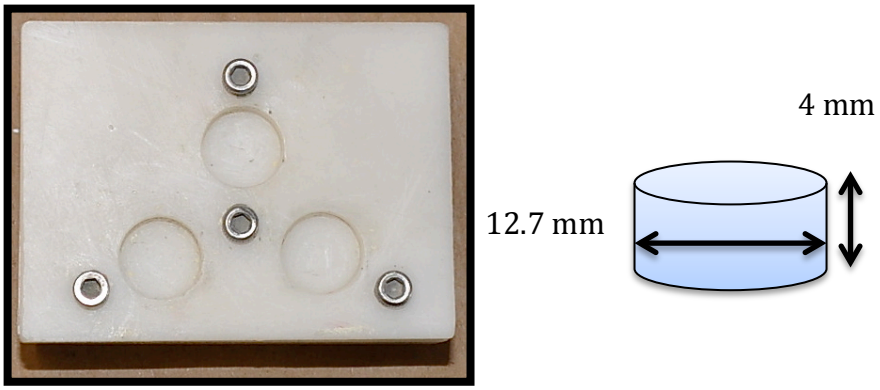


Figure 5-3: Mold used for setting time study.

Figure 5-4: Dial indicator set up used for the setting time test.

5.2.4. pH

The specimens of this test were obtained from a block of Teflon® mold that was 6 cm wide, 15 cm long, and 1.4 cm thick as shown in Figure 5-5(a). The mold consisted of 12 wells, each with a diameter of 15.4 mm and thickness of 2 mm (Figure 5-5(b).

(a) (b)

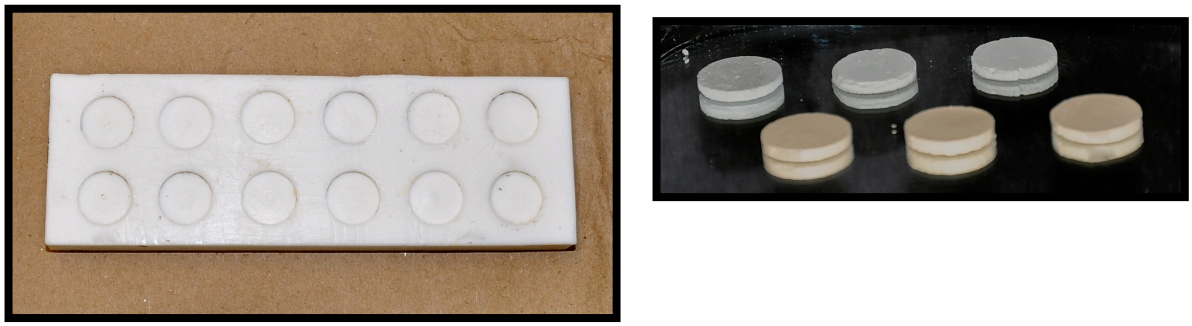


Figure 5-5: (a) Mold used for pH testing. (b) Samples used for pH test.

The mold containing the sample was stored for 4 hours in a leak-proof container covered with a moist paper towel in a 37º C oven and 100% humidity. Specimens were then transferred to 50 mL disposable tubes (Fischer Scientific, Springfield, NJ) filled with 20 mL of deionized water. Prior to measurement, the tubes were placed on a shaker for 5 minutes at 200 rpm (Gyrotory Shaker, New Brunswick Scientific, Edison, NJ).

Using pH meter (Accumet, Fischer Scientific) shown in Figure 5-6, the pH was measured at the desired time intervals. The pH meter was calibrated using buffer standard

solutions of pH 4.0, 7.0, and 12.46 (Thermo Fisher Scientific, Chelmsford, MA). The pH changes of the tested materials were recorded at a periods of 2 hours, 24 hours, 7 days, 21 days, and 28 days.

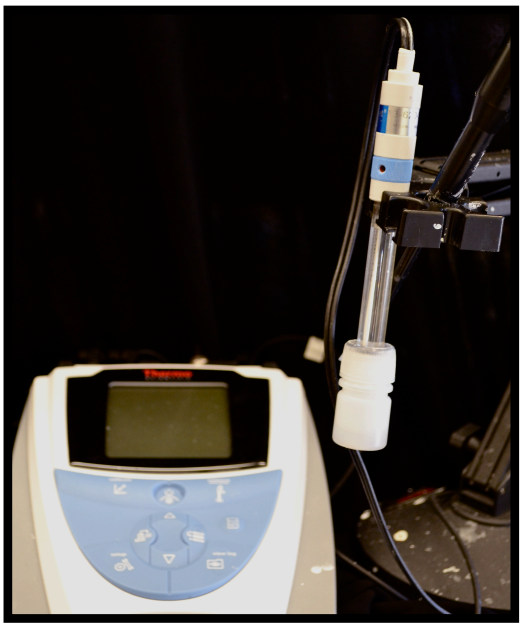


Figure 5-6: pH-meter.

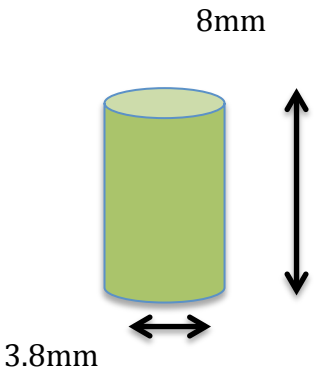
5.2.5 Compressive Strength

A modification of ISO specification 9917 (for water-based cement) was used for testing the compressive strength. After 24 hours, 7 days, 21 days and 28 days of storage in 37°C at 100% humidity, specimens were tested using a universal testing machine (Instron 5566A, Norwood, MA). A 1.0 kN load cell was used at a crosshead speed of 1 mm/min.

Specimen preparation was performed according to the same method described before in section 5.2.1. A Teflon® (6 cm × 15 cm × 1.4 cm) mold containing 12 wells was used in this test. Each well has a diameter of 3.8 mm and a depth of 8 mm (Figure 5-7 (a)). Cement was then compacted into each mold using a spatula and was further compacted using a dental plugger to ensure dense uniform samples with minimal porosity. Excess material was scraped off with the single-edge razor blade to leave a flat, uniform surface.

After the material was packed, the mold was placed for 24 hours in a leak-proof plastic container lined with moist paper towels at 37°C and 100% humidity. After that, the specimens were removed from the molds and visually examined for any air voids or chipped edges. All defective specimens were discarded, and a total of 10 accepted samples (Figure 5-7 (b)) were transferred to a disposable polystyrene dish (Fisher Scientific, Springfield, NJ), but kept in the same temperature and humidity conditions until tested. Immediately before testing, samples' diameters and lengths were measured with a digital caliper (SPI, Garden Grove, CA).

(a)



(b)

Figure 5-7: (a) Mold used for setting time testing. (b) Samples used for setting time.

5.2.6. Solubility

For the solubility test, 10 specimens of each material were prepared from the same mold that was used in the pH test. The mold containing the specimens was kept in 37ºC and 100% humidity for 24 hours to reach a constant weight. Initial weight (W_0) was recorded using an analytical balance. After that, samples were immersed in a petri dish containing 50 mL deionized water, and the plates were covered. Samples were removed, dehydrated in an oven at 37º C for 24 hours and weighed again at 24 hours, 7 days, 21 days, and 28 days (W_1, W_7, W_{21}, W_{28}). Immediately after being weighed, the samples were stored in a new petri dish containing 50 mL deionized water then dehydrated and weighed again at the following time interval. Solubility was calculated using the following formula:

$$Solubility = Weight\ difference / Initial\ weight \times 100$$

$$= [W_0 - (W_1, W_7, W_{21}, W_{28})] / W_0 \times 100$$

5.3 Statistical Analysis

Data were analyzed using the Statistical Analysis Software (SAS) program version 9.1.3. Descriptive results will be presented as mean value ± standard deviation. For multivariate analyses, data from the studies were analyzed for statistical significance with two-way analysis of variance (ANOVA). When significant differences were found ($p < 0.05$), multiple comparisons were done using the Tukey method to determine statistically significant differences between the groups' means.

A linear regression analysis was used for setting time, pH level, compressive strength, and solubility to predict indicators of higher overall scores for variables independently. Moreover, to identify which predictors (time and $CaCl_2$) have a greater impact on the overall dependent variable score, standardized beta coefficients were used.

RESULTS

6. RESULTS

6.1. Particle Size

The particle sizes of the Dycal® were examined first under the SEM and showed a range of < 10 μm. After that, the components of the rest experimental materials were examined under the SEM. These include the components of the EMTA (Type I Portland cement, bismuth oxide, and gypsum), the components of the BIE-containing materials (Type I Portland cement, bismuth oxide, and BIE material).

Except for the BIE material, the remaining tested materials showed a particle size of less than 10 μm. SEM images of Dycal®, Portland cement, bismuth oxide, and gypsum materials are shown in Figures 6-8 to 6-11, respectively.

The BIE material showed the largest particle sizes, ranging from 275 μm to 682 μm (Figure 6-12). Therefore, the material was subjected to further grinding using zirconia balls on the milling machine for 48 hours as previously described in section 5.2.1.2. After this process, the BIE demonstrated a particle size of less than 10 μm (Figure 6-13). All SEM images include measurements of at least three randomly selected particles from each material.

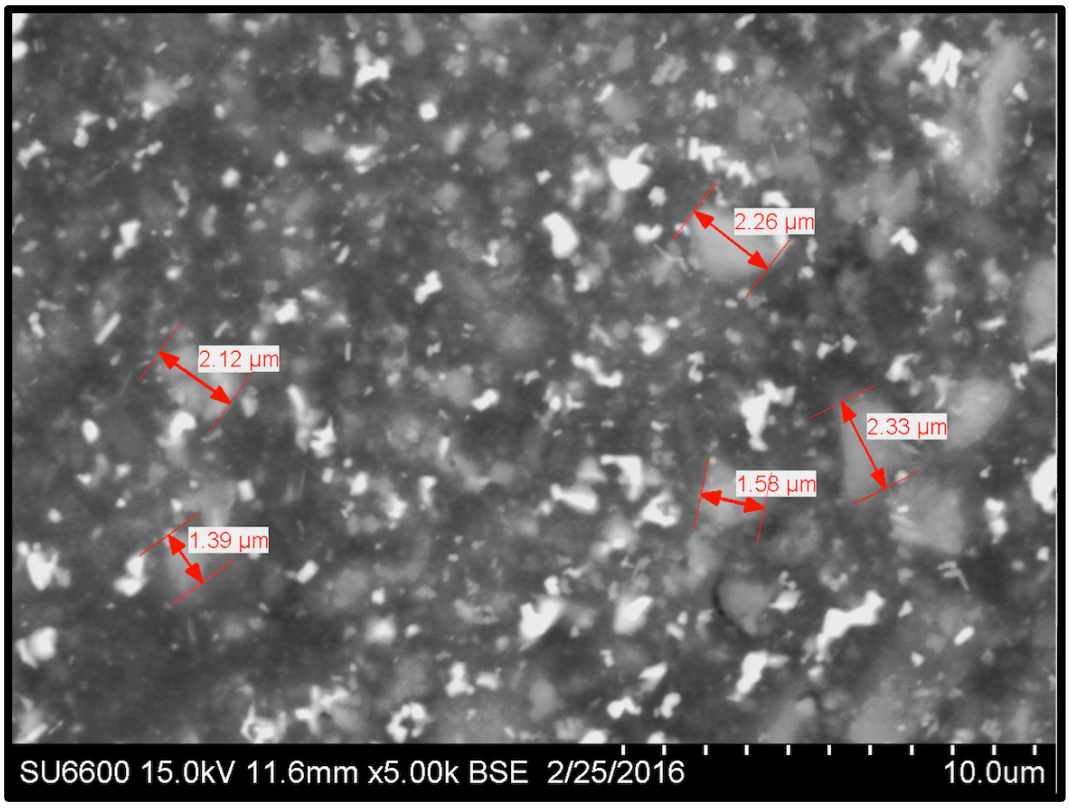


Figure 6-8: SEM images of Dycal®.

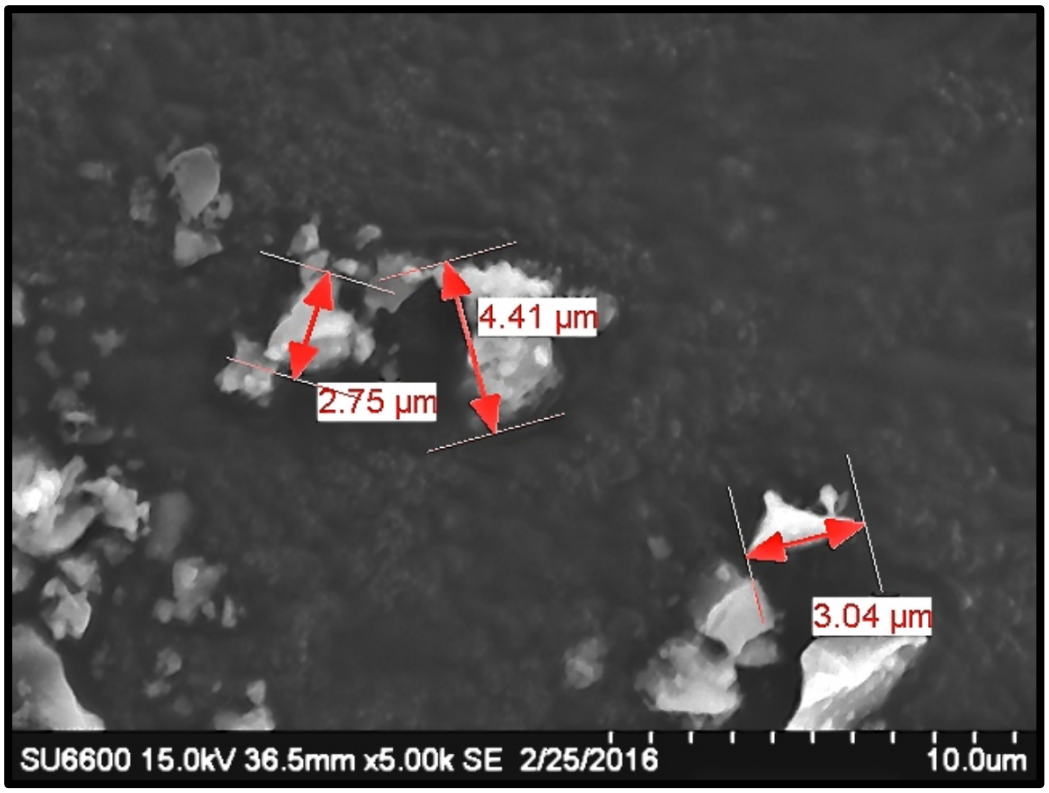


Figure 6-9: SEM images of Portland cement

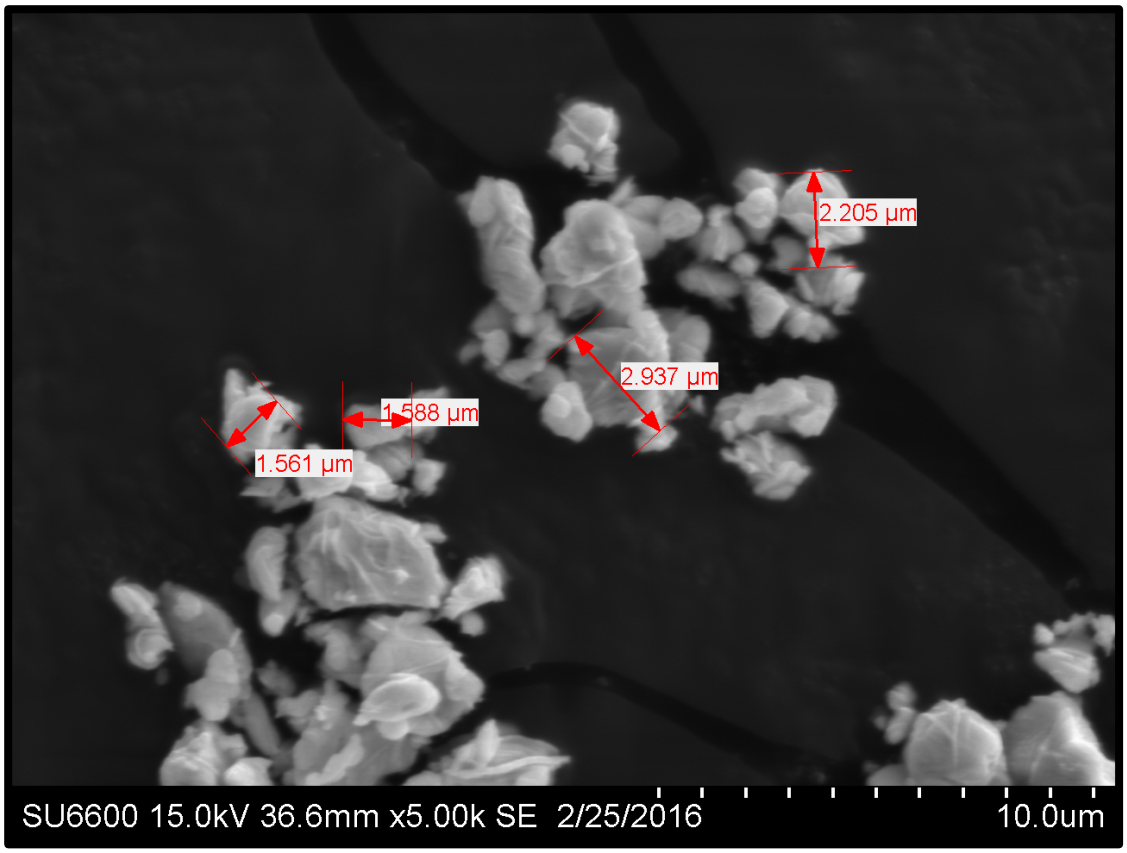


Figure 6-10: SEM images of bismuth oxide

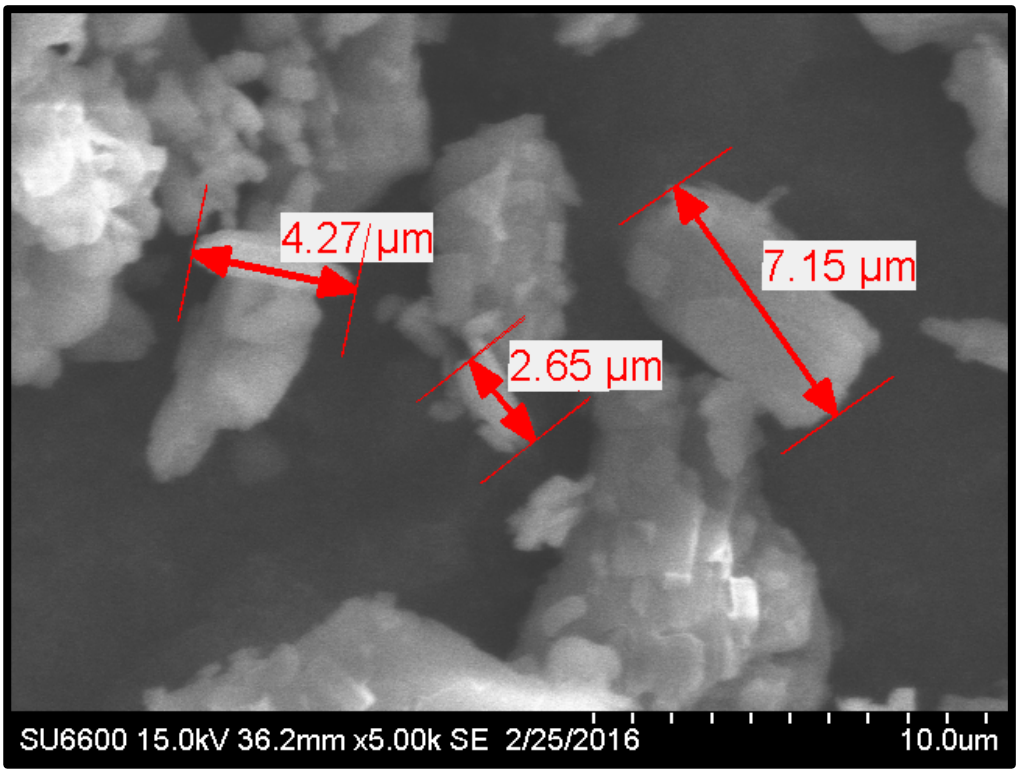


Figure 6-11: SEM images of gypsum

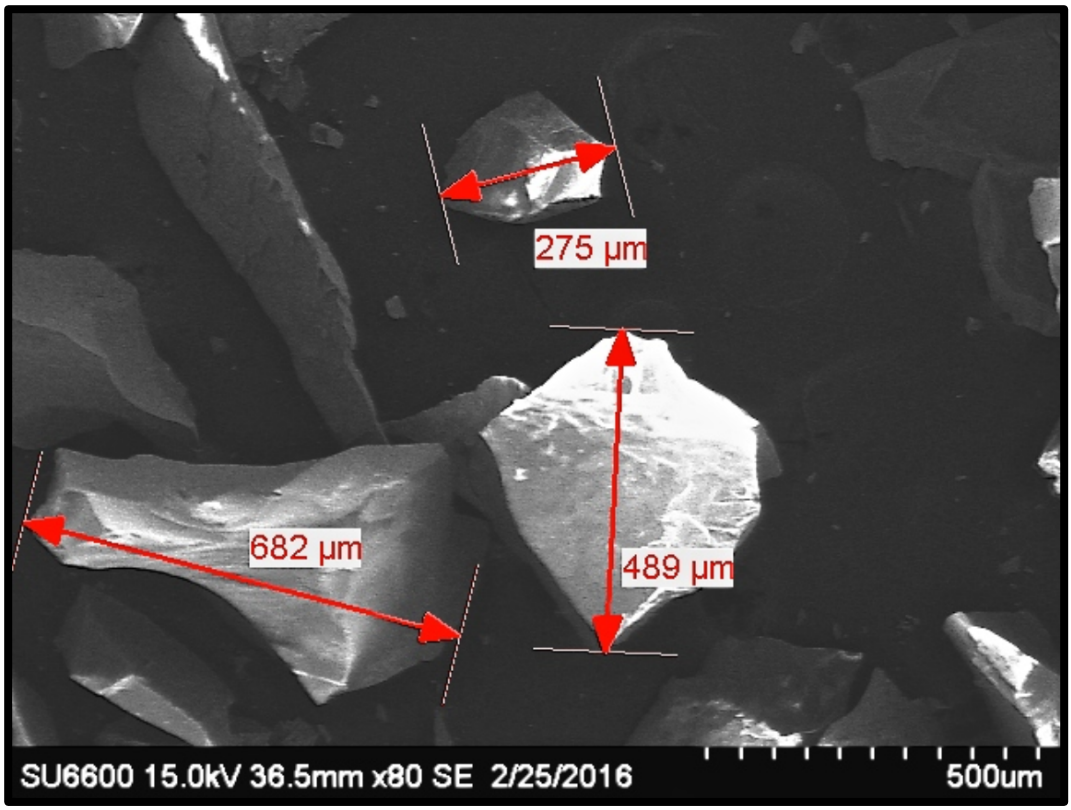


Figure 6-12: SEM images of BIE before grinding.

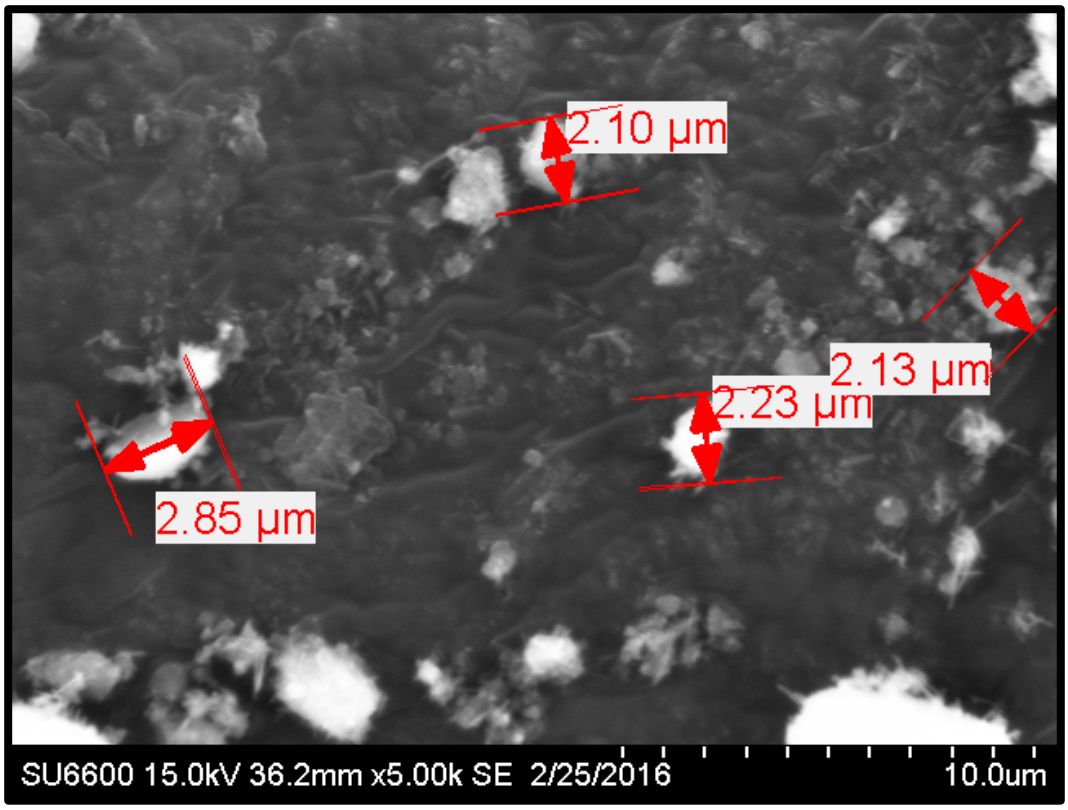


Figure 6-13: SEM images of BIE after grinding

6.2. Setting Time

The depths of penetration (mm) of the control group Dycal®, and experimental groups are shown in Appendix Tables 10-28 to 10-40. Experimental groups include the following:

1. EMTA
2. EMTA + 5% $CaCl_2$
3. EMTA + 10% $CaCl_2$
4. EMTA + 15% $CaCl_2$
5. 20% BIE
6. 20% BIE + 5% $CaCl_2$
7. 20% BIE + 10% $CaCl_2$
8. 20% BIE + 15% $CaCl_2$
9. 40% BIE
10. 40% BIE + 5% $CaCl_2$
11. 40% BIE + 10% $CaCl_2$
12. 40% BIE + 15% $CaCl_2$

A graphical presentation of the mean setting time of all material is shown in Figure 6-14.

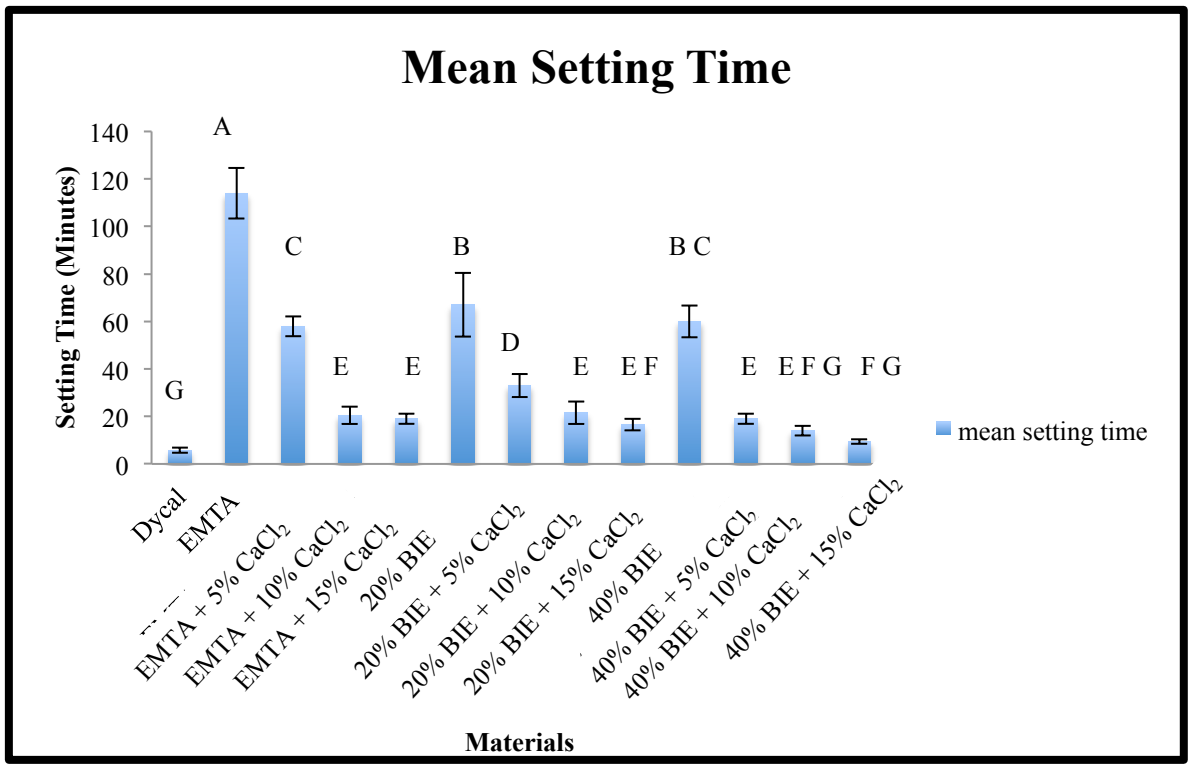


Figure 6-14: Mean setting time of all groups.

The results of the two-way ANOVA (Table 6-4) show that the setting time was statistically different among the groups ($p < 0.0001$). The setting time was statistically affected by the type of the material (Dycal®, EMTA, 20% BIE and 40% BIE) and the different concentrations of $CaCl_2$ (5%, 10%, and 15%). An interaction was also found between the two variables (materials and $CaCl_2$ concentrations).

The results of the mean setting time and the Tukey's multiple comparisons test of all tested materials are shown in Table 6-5. Dycal® showed significantly the shortest setting time (5.80 ± 1.13 minutes) among the groups, except for 40% BIE + 10% $CaCl_2$ (14.00 ± 2.10 minutes) and 40% BIE + 15% $CaCl_2$ (9.40 ± 0.96 minutes), where the difference was not significant ($p > 0.05$). On the other hand, EMTA showed the longest setting time (114.00 ± 10.74 minutes) at $p < 0.0001$.

The results of comparing the mean setting time of the materials without adding $CaCl_2$ (Dycal®, EMTA, 20% BIE, and 40% BIE) showed that Dycal® had statistically the shortest setting time (5.80 ± 1.13 minutes; $p <0.0001$), followed by 40% BIE then 20% BIE without significant difference between the setting time of both materials (60.00 ± 6.66, 67 ± 13.37 minutes; $p >0.05$). The EMTA material had the longest setting time (114.00 ± 10.74 minutes; $p <0.0001$).

Addition of calcium chloride had a substantial effect on setting time. It significantly decreased the setting time of the material when added in the range of 5% to 15%. However,

adding 15% of $CaCl_2$ did not significantly reduce the setting time compared to 10% of $CaCl_2$ when added to any material.

Table 6-4: Two-Way ANOVA of Setting Time.

Variable	Type lll Sum of Squares	df	Mean Square	F-Value	p-Value
Material (Dycal®, EMTA, 20% BIE and 40% BIE)	57079.65	3	19026.55	556.87	< 0.0001
$CaCl_2$ Concentration	80554.69	3	26851.56	785.90	< 0.0001
Material * $CaCl_2$ Concentration	10528.88	6	1754.81	51.33	< 0.0001

Table 6-5: Mean Setting Time of All Groups and Results of Tukey Multiple Comparisons Test.

Specimen (n=10)	Mean (Min)	SD	Min	Max	Tukey's Letter (¥)	p-value < 0.05
Dycal® (1)	5.80	1.13	4.00	8.00	G	1& (2*,3*,4*,5*,6*,7*,8*,9,10*, 11)
EMTA (2)	114.00	10.74	90.00	130.00	A	2& *(1,3,4,5,6,7,8,9,10,11,12, 13)
EMTA + 5% $CaCl_2$ (3)	58.00	4.216	50.00	60.00	C	3& (1*,2*,4*,5*,6,7*,8*,9,11*, 12*, 13*)
EMTA + 10% $CaCl_2$ (4)	20.50	3.689	15.00	30.00	E	4 & (1*,2*, 3*,6*,7,10*,13)
EMTA + 15% $CaCl_2$ (5)	19.00	2.108	15.00	20.00	E	5 & (1*,2*,3*,6*,7*,10*,13)
20% BIE (6)	67.00	13.37	50.00	90.00	B	6 & (1*,2*,3,4*,5*,7*,8*,9*,11*, 12*, 13*)
20% BIE + 5% $CaCl_2$ (7)	33.00	4.83	30.00	40.00	D	7 & (1*,2*,3*,4,5*,6*,8,9*,10*, 11*,12*,13*)
20% BIE + 10% $CaCl_2$ (8)	22.50	5.400	15.00	30.00	E	8 & (1*,2*,3*,6,*7,10*,13)
20% BIE + 15% $CaCl_2$ (9)	16.50	2.41	15.00	20.00	E F	9 & (1,2*,3*,6*,7*,10*)
40% BIE (10)	60.00	6.66	50.00	70.00	B C	10 & *(1,2,4,5,7,8,9,11,12,13)
40% BIE + 5% $CaCl_2$ (11)	19.00	2.10	15.00	20.00	E	11 & (1,2*,3*,6*,7,*10*,13)
40% BIE + 10%$CaCl_2$ (12)	14.00	2.10	10.00	15.00	E F G	12 & *(2,3,6,7,10)
40% BIE + 15% $CaCl_2$ (13)	9.40	0.96	8.00	10.00	F G	13 & (2*,3*,4,5,6*,7*,8,10*,11)

* $p < 0.0001$

(¥) Materials with the same letters were not statistically significant ($p > 0.05$)

A linear regression model predicting the odds of having lower setting times based on $CaCl_2$ concentrations was performed (Table 6-6). The effect of $CaCl_2$ accounted for 36% of the variability in the overall setting time score (Adjusted $R^2 = 0.36$, $p < 0.0001$).

Table 6-6: Linear Regression Model Predicting Lower Setting Time

Variable	Estimate	p-value
$CaCl_2$ concentration	16.187	< 0.0001

This model showed that each unit (5%) increase in the concentration of $CaCl_2$ resulted in a setting time decrease of 16.187 minutes, after controlling of other variables.

6.3. pH

The mean pH changes of each tested material solutions over time intervals (2 hours, and 1, 7, 21, and 28 days) are presented in Figures 6-15 to 6-18. The pH changes of all materials by time intervals are presented in Appendix Table 10-41 through 10-45, respectively.

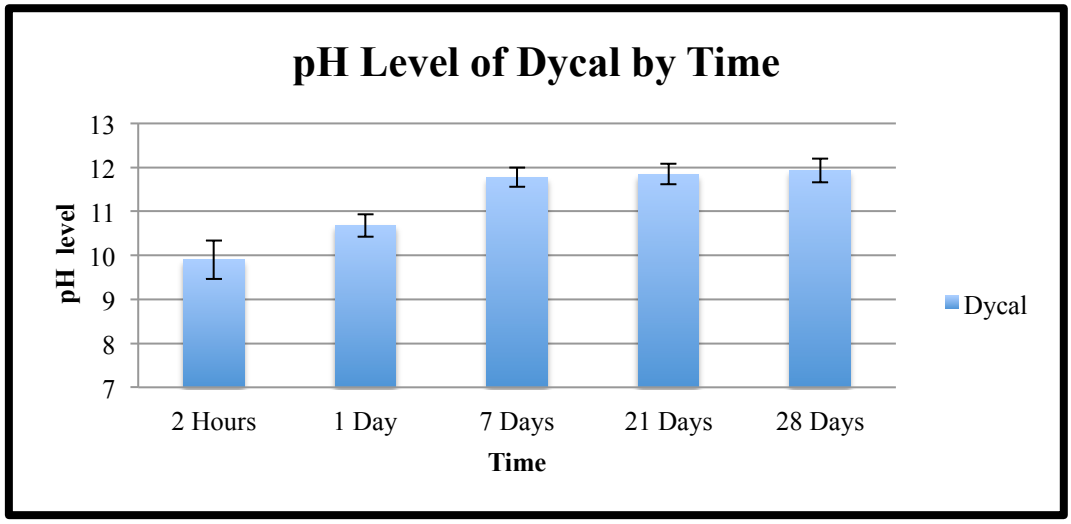


Figure 6-15: Mean pH levels of Dycal® over time intervals.

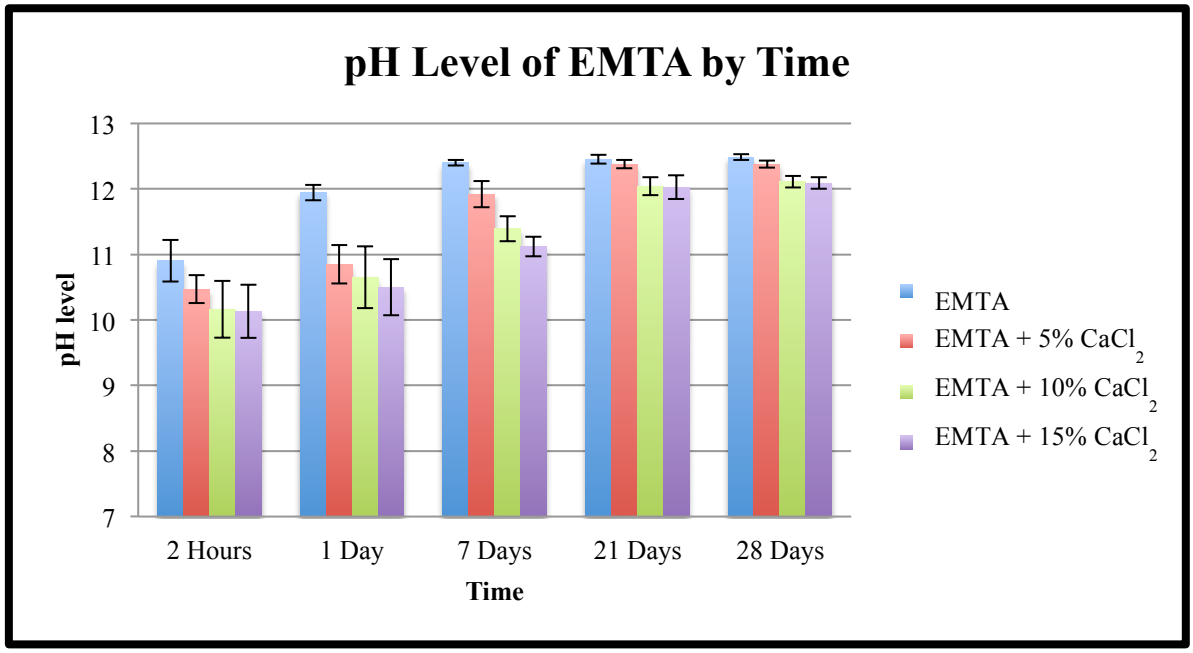


Figure 6-16: Mean pH level of EMTA, EMTA + (5%, 10%, and 15%) $CaCl_2$ over time intervals.

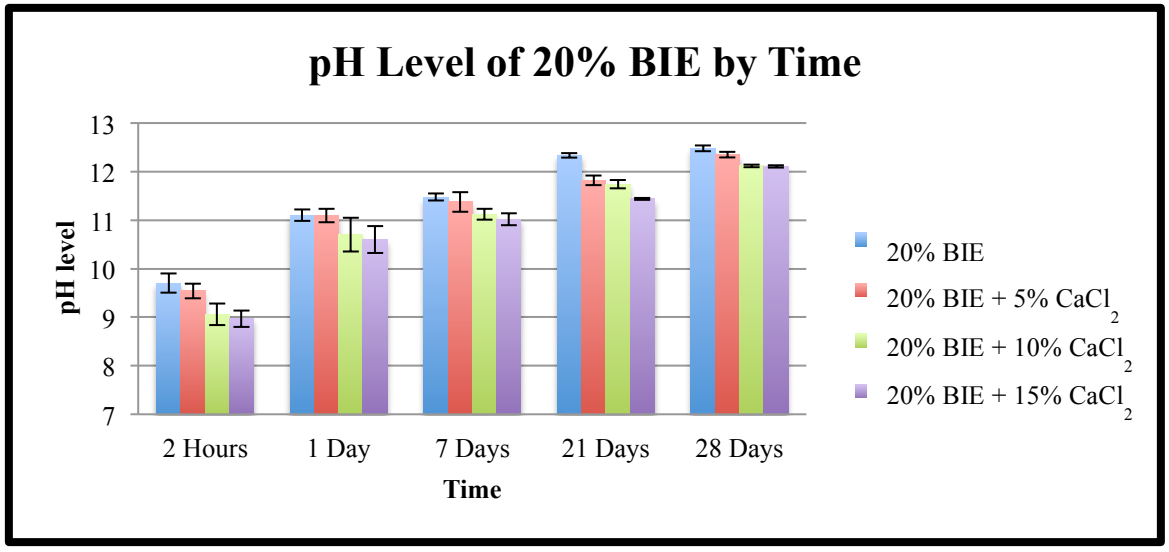


Figure 6-17: Mean pH level of 20% BIE and 20% BIE + (5%, 10%, and 15%) $CaCl_2$ over time intervals.

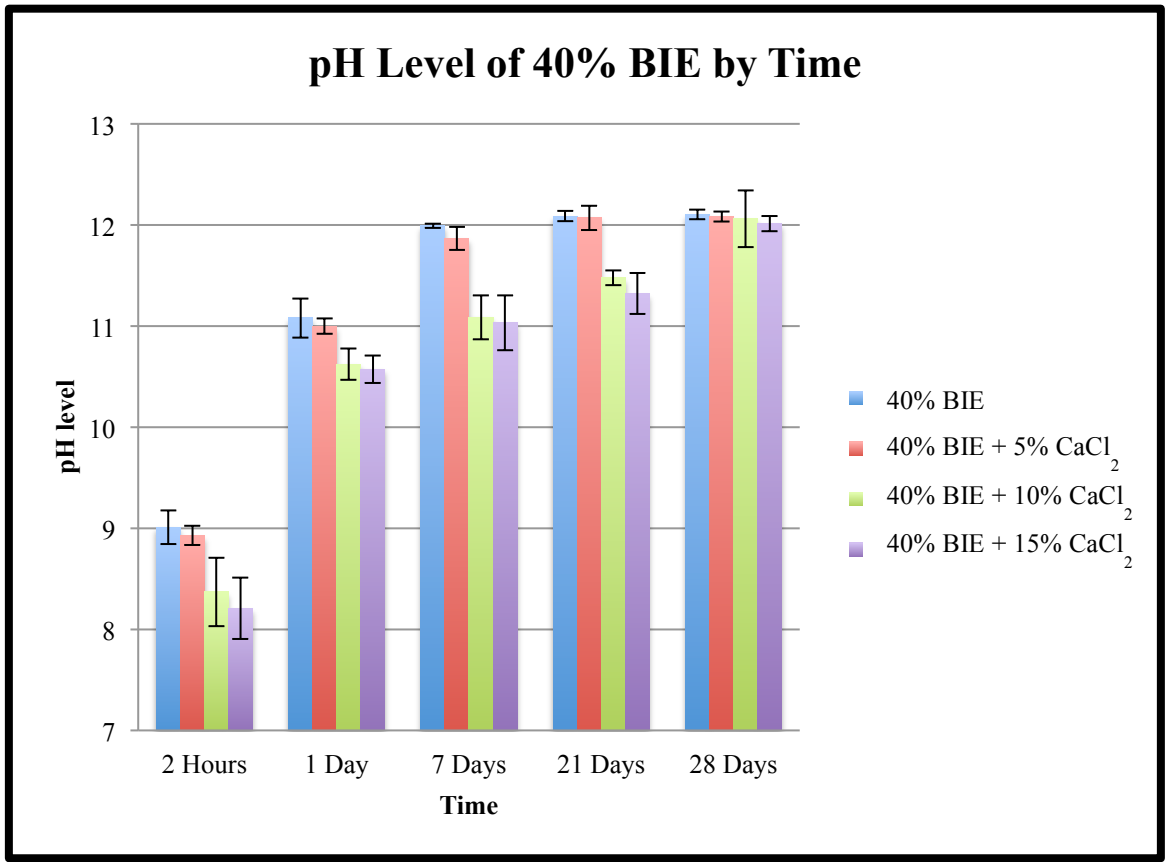


Figure 6-18: Mean pH level of 40% BIE and 40% BIE + (5%, 10%, and 15%) $CaCl_2$ over time intervals.

The results showed that the pH levels of equilibrated solutions for all tested materials increased with time. Adding calcium chloride decreased the pH level at the beginning, however, its effect diminished over time.

The results of two-way ANOVA (Table 6-7) showed that the pH level was statistically different among the groups ($p < 0.0001$). The pH level is statistically affected by the type of the material (Dycal®, EMTA, 20% BIE and 40% BIE), the different concentrations of $CaCl_2$ (5%, 10%, and 15%) and the different time intervals (2 hours, and 1, 7, 21, and 28 days). An interaction was also found between the three variables (material, $CaCl_2$ concentrations and time).

From Table 6-8 to 6-12, the mean pH levels of all tested materials and Tukey's multiple comparison test are presented at each time interval. A graphical presentation of the pH changes of all tested materials at each time intervals are presented in Figures 6-19 to 6-23.

At all time intervals the pH level of EMTA material was statistically the highest among the tested groups ($p <0.0001$). At 2 hours, the pH level of Dycal® (9.90 ± 0.43) was comparable to EMTA + 10% $CaCl_2$ (10.16 ± 0.43), EMTA + 15% $CaCl_2$ (10.13 ± 0.4), 20% BIE (9.70 ± 0.20), and 20% BIE + 5% $CaCl_2$ (9.54 ± 0.15) without significant difference at $p > 0.05$. The initial pH level, at 2 hours, of the 40% BIE material (9.01 ± 0.16) was lower than that of Dycal®, EMTA, and 20% BIE at $p < 0.0001$.

For materials without $CaCl_2$, at 24 hours, 7, 21, and 28 days, the EMTA material showed the highest pH level followed by the 20% BIE, then the 40% BIE, and Dycal® material showed the lowest pH level. At 28 days, the pH level of Dycal® (11.93 ± 0.27) was comparable to the pH level of 40% BIE (12.10 ± 0.04) with $p > 0.05$. After the addition of $CaCl_2$, the pH level of all materials decreased, with no significant difference between adding 10% and 15% $CaCl_2$ to any tested materials ($p > 0.05$).

Table 6-7: Two-Way ANOVA of pH Level

Variable	Type lll Sum of Squares	df	Mean Square	F-Value	p-Value
Material (Dycal®, EMTA, 20% BIE and 40% BIE)	33.74	3	11.24	268.39	< 0.0001
$CaCl_2$ Concentration	52.27	3	17.42	415.82	< 0.0001
Time	591.70	4	147.92	3529.88	< 0.0001
Material * $CaCl_2$ Concentration * Time	72.32	54	1.33	31	< 0.0001

Table 6-8: Mean pH of All Groups and Results of Tukey Multiple Comparisons Test at 2 Hours.

Specimen (n=10)	Mean	SD	Min	Max	Tukey's Letter (¥)	p < 0.05
Dycal® (1)	9.90	0.43	9.10	10.70	C E D	1& (2*,3, 8*,9*,10*,11*,12*,13*)
EMTA (2)	10.90	0.31	10.25	11.22	A	2& (1*, 3,4*,5*,6*,7*,8*,9*,10*,11*,12*,13*)
EMTA + 5% $CaCl_2$ (3)	10.47	0.21	10.20	10.80	B	3 & (1,2,6*,7*,8*,9*,10*,11*,12*,13*)
EMTA + 10% $CaCl_2$ (4)	10.16	0.43	9.40	10.66	B C	4 & (2*,6,7,8*,9*,10*,11*,12*13*)
EMTA + 15% $CaCl_2$ (5)	10.13	0.40	9.40	10.66	B C D	5 & (2*,7,8*,9*,10*,11*,12*,13*)
20% BIE (6)	9.70	0.20	9.30	9.90	E D	6 & (2*,3*,4,8,9*,10*,11,*12,*13*)
20% BIE + 5% $CaCl_2$ (7)	9.54	0.15	9.30	9.80	E	7& (2*,3*,4,5,8,9,10,11,12*,13*)
20% BIE + 10% $CaCl_2$ (8)	9.06	0.22	8.80	9.40	F	8& (1*,2*,3*,4*,5*,6,7,12*,13*)
20% BIE + 15% $CaCl_2$ (9)	8.97	0.17	8.80	9.30	F	9& (1*,2*,3*,4*,5*,6*,7,12,13*)
40% BIE (10)	9.01	0.16	8.80	9.30	F	10& (1*,2*,4*,5*,6*,7,12,13*)
40% BIE + 5% $CaCl_2$ (11)	8.92	0.09	8.80	9.10	F	11& (1*,2*,3*,4*,5*,6*,7,12,13*)
40% BIE + 10%$CaCl_2$ (12)	8.37	0.33	7.88	8.70	G	12& (1*,2*,3*,4*,5*,6*,7*,8*,9,10,11)
40% BIE + 15% $CaCl_2$ (13)	8.20	0.30	7.880	8.55	G	13& *(1,2,3,4,5,6,7,8,9,10,11)

* $p < 0.0001$

(¥) Materials with the same letters were not statistically significant ($p > 0.05$)

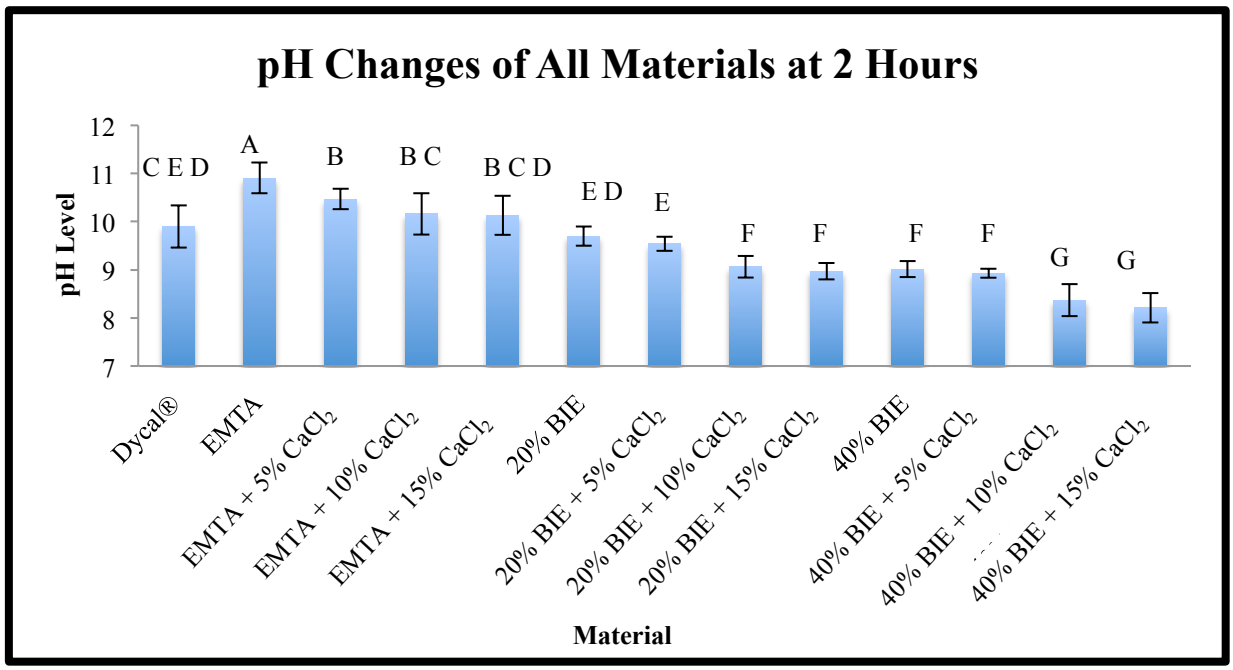


Figure 6-19: Mean pH of all groups and ANOVA at 2 hours

Table 6-9: Mean pH of All Groups and Results of Tukey Multiple Comparisons Test at 24 Hours.

Specimen (n=10)	Mean	SD	Min	Max	Tukey's Letter (¥)	p-value < 0.05
Dycal® (1)	10.68	0.25	10.40	11.10	D	1& (2*,6,7,10*,11*)
EMTA (2)	11.94	0.19	11.75	12.09	A	2& *(1,3,4,5,6,7,8,9,10,11,12,13)
EMTA + 5% $CaCl_2$ (3)	10.85	0.29	10.30	11.20	B C D	3 & *(2,10,11)
EMTA + 10% $CaCl_2$ (4)	10.65	0.47	9.86	11.30	D	4 & (2*,6,7,10*,11*)
EMTA + 15% $CaCl_2$ (5)	10.50	0.43	9.40	10.99	D	5 & (2*,6,7, 10*,11*)
20% BIE (6)	11.10	0.11	10.90	11.20	B	6 & (1,2*,4,5,8,9,10*,11,12,13)
20% BIE + 5% $CaCl_2$ (7)	11.09	0.13	10.80	11.20	B C	7& (1,2*,4,5,9,10*,11,12,13)
20% BIE + 10% $CaCl_2$ (8)	10.70	0.34	9.90	11.10	C D	8& (2*,6,10*,11*)
20% BIE + 15% $CaCl_2$ (9)	10.59	0.28	9.90	10.88	D	9& (2*,6,7,10*,11*)
40% BIE (10)	11.08	0.19	11.50	12.10	B	10& *(1,2,3,4,5,6,7,8,9,12,13)
40% BIE + 5% $CaCl_2$ (11)	11.00	0.07	11.46	11.69	B	11& (1*,2,*3*,4*,5*,6,7,8*,9*,12*,13*)
40% BIE + 10%$CaCl_2$ (12)	10.62	0.15	10.40	10.88	D	12& (2*,6,7,10*,11*)
40% BIE + 15% $CaCl_2$ (13)	10.57	0.13	10.40	10.80	D	13& (2*,6,7, 10*,11*)

* $p < 0.0001$

(¥) Materials with the same letters were not statistically significant ($p > 0.05$).

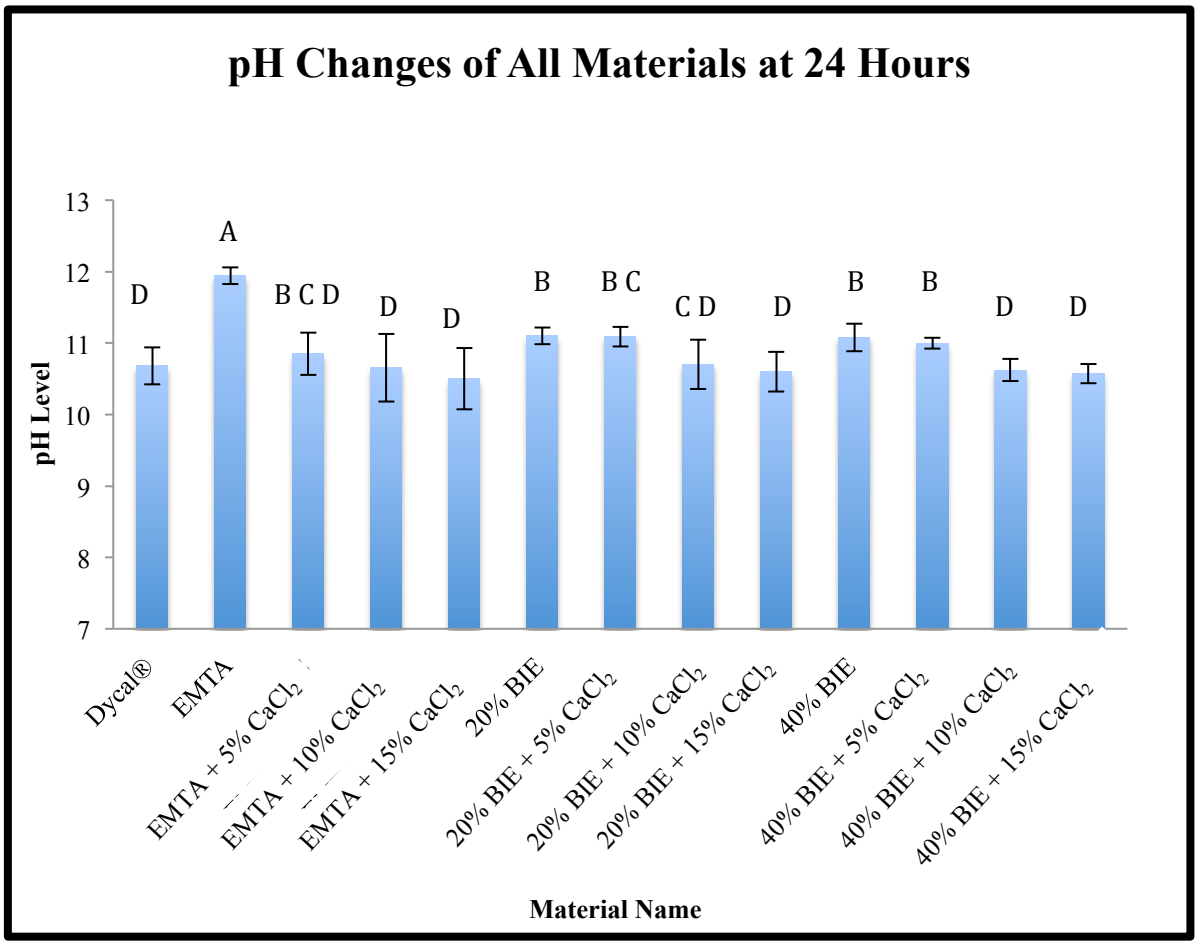


Figure 6-20: Mean pH of all groups and ANOVA at 24 hours.

Table 6-10: Mean pH of All Groups and Results of Tukey Multiple Comparisons Test at 7 Days.

Specimen (n=10)	Mean	SD	Min	Max	Tukey's Letter (¥)	p-value < 0.05
Dycal® (1)	11.78	0.22	11.40	12.00	B	1& (2*,4*,5*,8*,9*,12*,13*)
EMTA (2)	12.39	0.04	12.37	12.48	A	2& *(1,3,4,5,6,7,8,9,10,11,12,13)
EMTA + 5% $CaCl_2$ (3)	11.92	0.20	11.60	12.17	B	3& *(2,4,5,8,9,12,13)
EMTA + 10% $CaCl_2$ (4)	11.39	0.19	11.00	11.60	C	4& (1*,2*,3*,5,8,9,10*,11*,12,13)
EMTA + 15% $CaCl_2$ (5)	11.12	0.14	10.90	11.30	D	5& (1*,2*,3*,4,6,7,10*,11*)
20% BIE (6)	12.00	0.07	11.40	11.60	B	6& (2*,5,8,9*,12*,13*)
20% BIE + 5% $CaCl_2$ (7)	11.91	0.20	10.98	11.67	B	7& (2*,5,8,9,12,13)
20% BIE + 10% $CaCl_2$ (8)	11.12	0.11	11.00	11.28	D	8& (1*,2*,3*,4,6,7,10*,11*)
20% BIE + 15% $CaCl_2$ (9)	11.02	0.12	10.88	11.22	D	9& (1*,2*,3*,4,6*,7*,10*,11*)
40% BIE (10)	11.99	0.02	11.97	12.05	B	10& *(2,4,5,8,9,12,13)
40% BIE + 5% $CaCl_2$ (11)	11.86	0.11	11.60	12.02	B	11& *(2,4,5,8,9,12,13)
40% BIE + 10%$CaCl_2$ (12)	11.08	0.21	10.85	11.64	D	12& (1*,2*,3*,4,6*,7,10*,11*)
40% BIE + 15% $CaCl_2$ (13)	11.03	0.27	10.70	11.40	D	13& (1*,2*,3*,4,6*,7,10*,11*)

* $p < 0.0001$

(¥) Materials with the same letters were not statistically significant ($p > 0.05$)

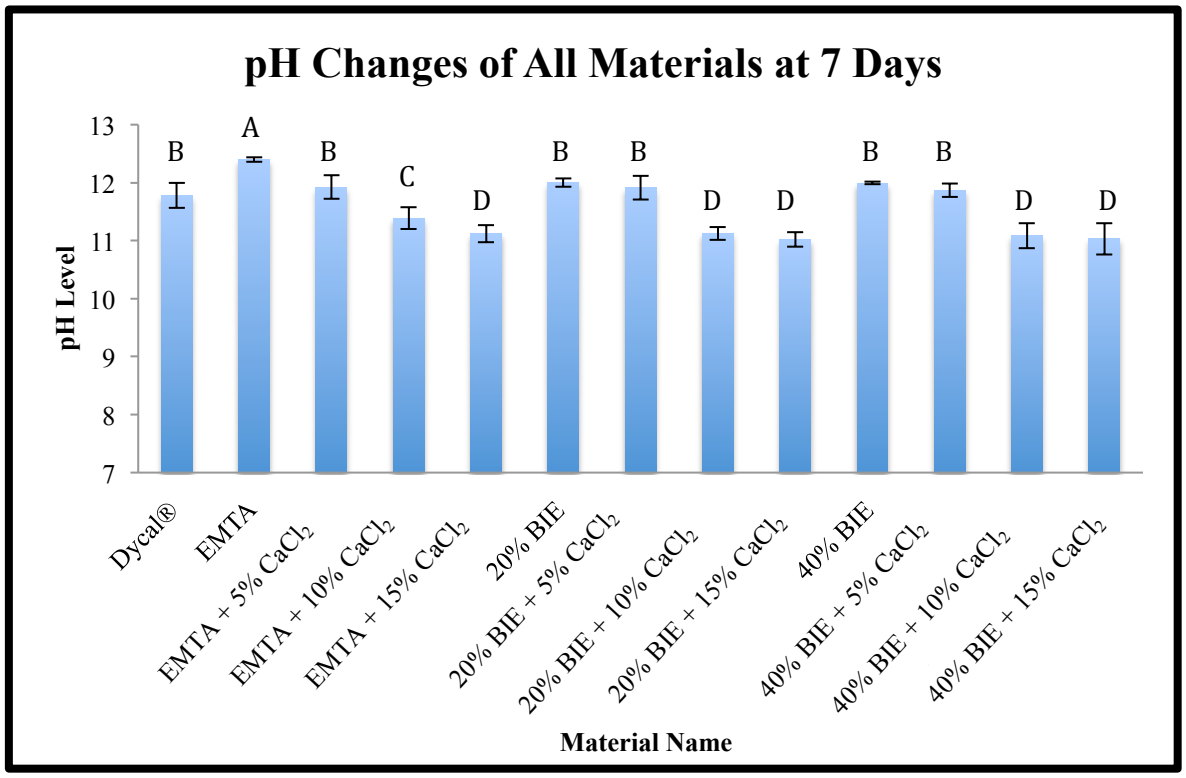


Figure 6-21: Mean pH of all groups and ANOVA at 7 days.

Table 6-11: Mean pH of All Groups and Results of Tukey Multiple Comparisons Test at 21 Days.

Specimen (n=10)	Mean	SD	Min	Max	Tukey's Letter (¥)	p < 0.05
Dycal® (1)	11.85	0.23	11.40	12.20	C D	1& (2*,3*,4,7*,9*,10,11,12*,13*)
EMTA (2)	12.45	0.06	12.31	12.54	A	2& *(1,4,5,6,8,9,10,11,12,13)
EMTA + 5% $CaCl_2$ (3)	12.37	0.06	12.29	12.50	A	3& *(1,4,5,6,8,9,10,11,12,13)
EMTA + 10% $CaCl_2$ (4)	12.04	0.13	11.80	12.20	B	4& (1,2*,3*,6,7*,8*,9*,12*,13*)
EMTA + 15% $CaCl_2$ (5)	12.02	0.18	11.64	12.20	B C	5& *(2,3,6,7,8,9,12,13)
20% BIE (6)	12.34	0.05	12.25	12.41	A	6& (2*,3*,4,5,7*,8,9*,10,11,12*, 13*)
20% BIE + 5% $CaCl_2$ (7)	11.82	0.10	11.70	12.00	D	7& (1*,4*,5*,6*,8*,9*,10,11,12*, 13*)
20% BIE + 10% $CaCl_2$ (8)	11.74	0.08	11.65	11.92	D	8& (2*,3*,4*,5*,6*,7*,9*,10*,11*, 12,13*)
20% BIE + 15% $CaCl_2$ (9)	11.43	0.02	11.40	11.47	E	9& *(1,2,3,4,5,6,7,8,10,11)
40% BIE (10)	12.09	0.05	12.02	12.18	B	10& (1,2*,3*,6,7,8*,9*,12*,13*)
40% BIE + 5% $CaCl_2$ (11)	12.07	0.12	11.89	12.28	B	11& (1,2*,3*,6,7,8*,9*,12*,13*)
40% BIE + 10%$CaCl_2$ (12)	11.47	0.07	11.40	11.60	E	12& (1*,2*,3*,4*,5*,6*,7*,8,10*, 11*)
40% BIE + 15% $CaCl_2$ (13)	11.32	0.20	11.00	11.60	E	13& *(1,2,3,4,5,6,7,8,10,11)

* $p < 0.0001$

(¥) Materials with the same letters were not statistically significant ($p > 0.05$)

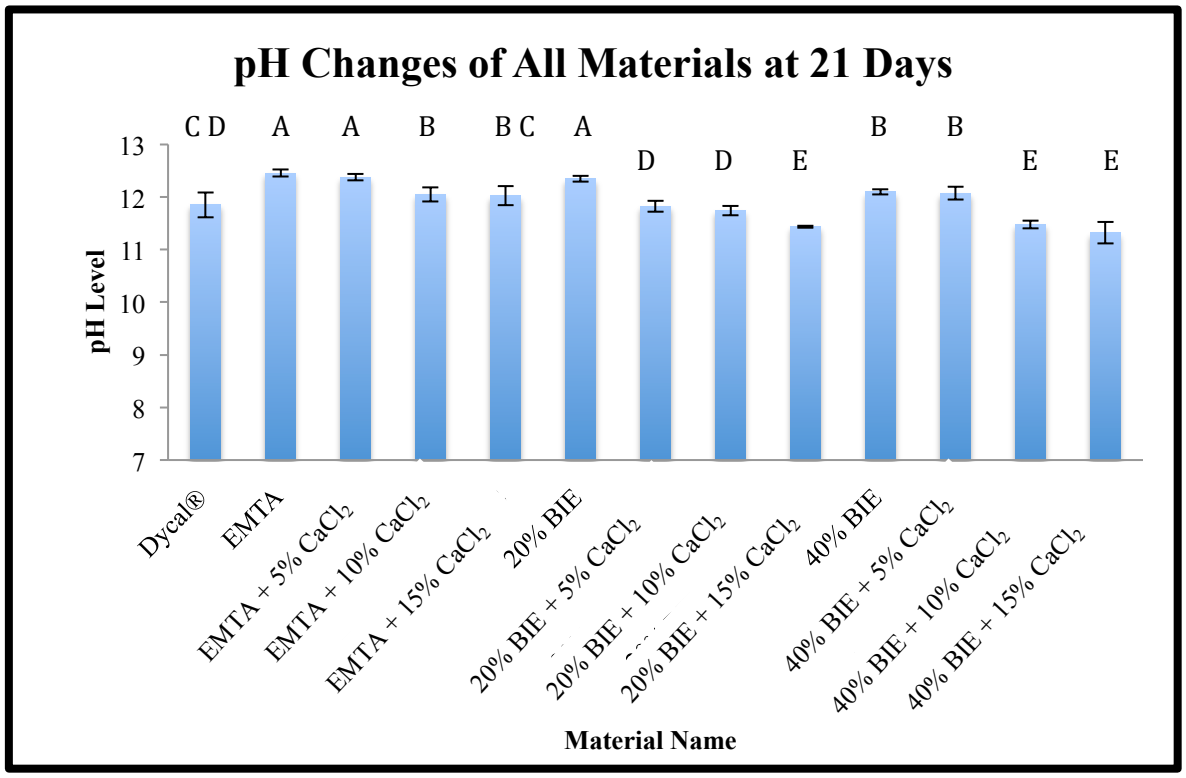


Figure 6-22: Mean pH of all groups and ANOVA at 21 days.

Table 6-12: Mean pH of All Groups and Results of Tukey Multiple Comparisons Test at 28 days

Specimen (n=10)	Mean	SD	Min	Max	Tukey's Letter (¥)	p-value <0.05
Dycal® (1)	11.93	0.27	11.60	12.50	B C	1& *(2,3,6,7)
EMTA (2)	12.48	0.04	12.43	12.59	A	2& *(1,4,5,8,9,10,11,12,13)
EMTA + 5% $CaCl_2$ (3)	12.38	0.05	12.31	12.50	A	3& (1*,4,5*,8,9,10,11*,12*,13*)
EMTA + 10% $CaCl_2$ (4)	12.11	0.08	12.00	12.20	B C	4& (2*,3,6*,7)
EMTA + 15% $CaCl_2$ (5)	12.09	0.08	12.00	12.20	B C	5& (2*,3*,6*,7)
20% BIE (6)	12.47	0.05	12.40	12.57	A	6& *(1,4,5,8,9,10,11,12,13)
20% BIE + 5% $CaCl_2$ (7)	12.35	0.05	12.27	12.46	A	7& (1*,4,5,8,9,10,11,12*,13*)
20% BIE + 10% $CaCl_2$ (8)	12.11	0.03	12.08	12.17	B	8& (2*,3,6*,7)
20% BIE + 15% $CaCl_2$ (9)	12.10	0.02	12.02	12.15	B C	9& (2*,3,6*,7)
40% BIE (10)	12.10	0.04	12.40	12.20	B C	10& (2*,3,6*,7)
40% BIE + 5% $CaCl_2$ (11)	12.08	0.05	12.02	12.18	B C	11& (2*,3*,6*,7)
40% BIE + 10%$CaCl_2$ (12)	12.06	0.28	11.28	12.22	B C	12& *(2,3,6,7)
40% BIE + 15% $CaCl_2$ (13)	12.01	0.07	11.97	12.22	B C	13& *(2,3,6,7)

* $p < 0.0001$

(¥) Materials with the same letters were not statistically significant ($p > 0.05$)

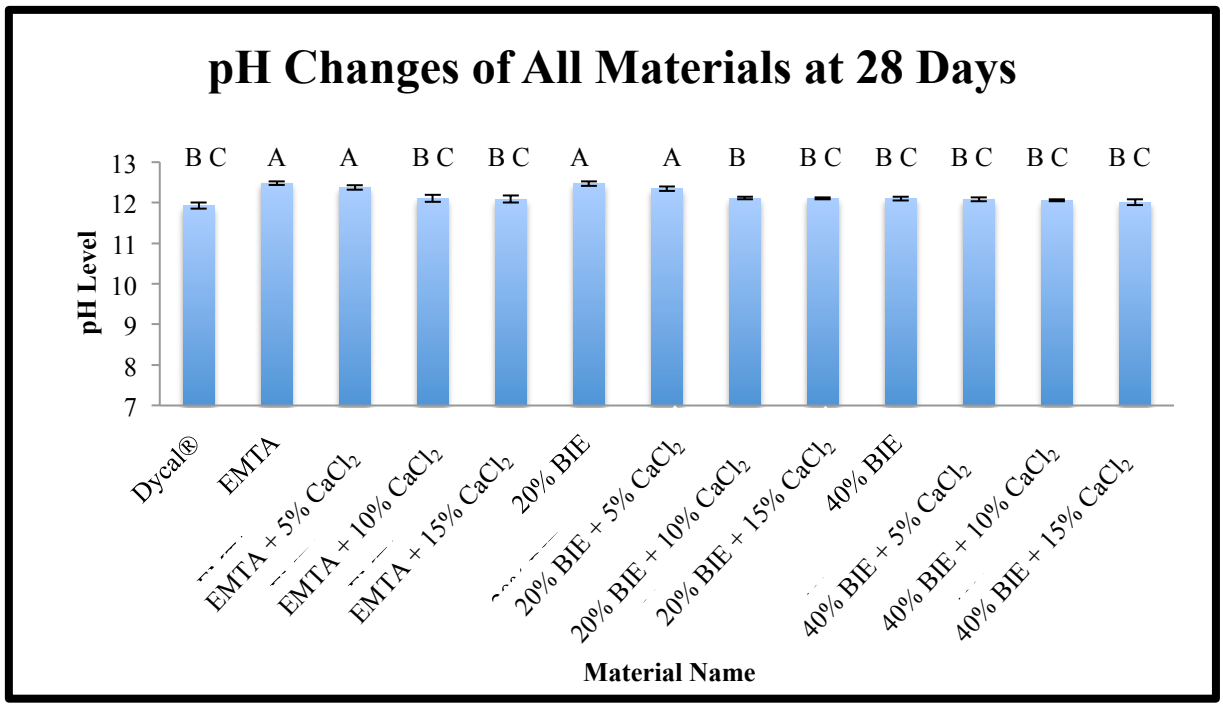


Figure 6-23: Mean pH of all groups and ANOVA at 28 days

A linear regression model predicting the odds of having higher pH levels based on $CaCl_2$ concentrations and time intervals was performed (Table 6-13). The effect of $CaCl_2$ and time accounted for 73% of the variability in the overall pH level (Adjusted $R^2 = 0.73$, $p < 0.0001$).

This model shows that each unit (5%) increase in the concentration of $CaCl_2$, resulted in a decrease in the pH level by 0.226 degrees. Each increase in the time interval resulted in an increase of the pH level by 0.632 degrees, after controlling for other variables.

Table 6-13: Linear Regression Model Predicting Higher pH Level.

Variable	Estimate	p-value
$CaCl_2$ concentration	-0.226	< 0.0001
Time	0.632	< 0.0001

6.4. Compressive Strength

The raw data of compressive strength of all tested materials vs. time are presented in Appendix Tables 10-46 through 10-58. The mean compressive strengths of each tested material over time intervals (day 1, 7, 21, and 28) are presented in Figures 6-24 to 6-27. The results revealed that the compressive strength of all tested materials increased with time, and adding $CaCl_2$ decreased the compressive strength of the material.

The results of the two-way ANOVA (Table 6-14) showed that the compressive strength was statistically different among the groups ($p < 0.0001$). The compressive strength was statistically affected by type of material (Dycal®, EMTA, 20% BIE, and 40% BIE), the different concentrations of $CaCl_2$ and the different time intervals. An interaction was also found between the three variables (material, $CaCl_2$ concentrations, and time).

The mean compressive strength of all tested materials vs. time (1, 7, 21, and 28 days), and the statistical analysis of two-way ANOVA at $p < 0.05$ and Tukey's multiple comparisons test are presented at each time interval from Table 6-15 to 6-18. A graphical representation of the mean compressive strengths of all tested materials vs. time (1, 7, 21, and 28 days) is presented from Figures 6-28 to 6-31.

At all intervals, Dycal® showed the lowest compressive strength material, and 40% BIE showed the highest. The difference in strength between Dycal® and the rest of the tested materials was statistically significant at $p < 0.0001$ at 1, 7, and 21 days. At 28 days the

compressive strength of the Dycal material showed no significant difference with EMTA + 10% and 15% $CaCl_2$ ($p > 0.05$). Adding any concentration of $CaCl_2$ (5%, 10% or 15%) to EMTA, 20% BIE or 40% BIE inversely affected its strength during all time intervals. However, this decline in strength was statistically significant only when 10% or 15% of $CaCl_2$ were added compared to same material without $CaCl_2$ ($p < 0.0001$).

There was no significant difference in the compressive strength of same materials with 10% $CaCl_2$ and 15% $CaCl_2$ ($p > 0.05$) at all time intervals.

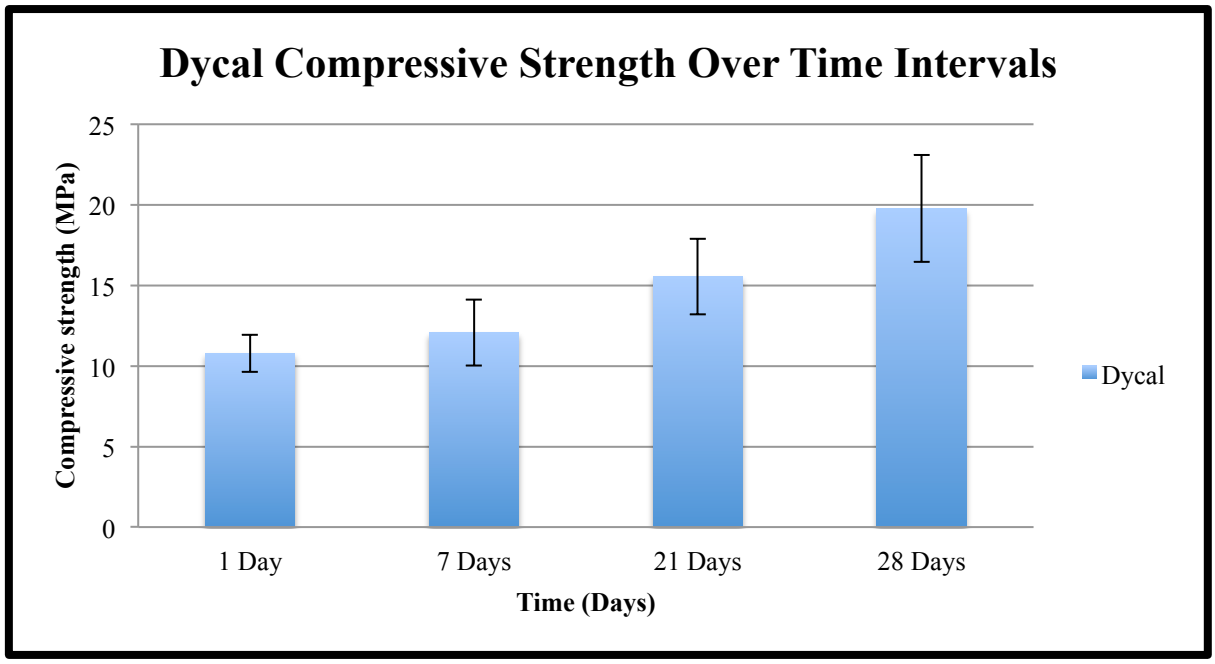


Figure 6-24: Dycal® compressive strength over time intervals.

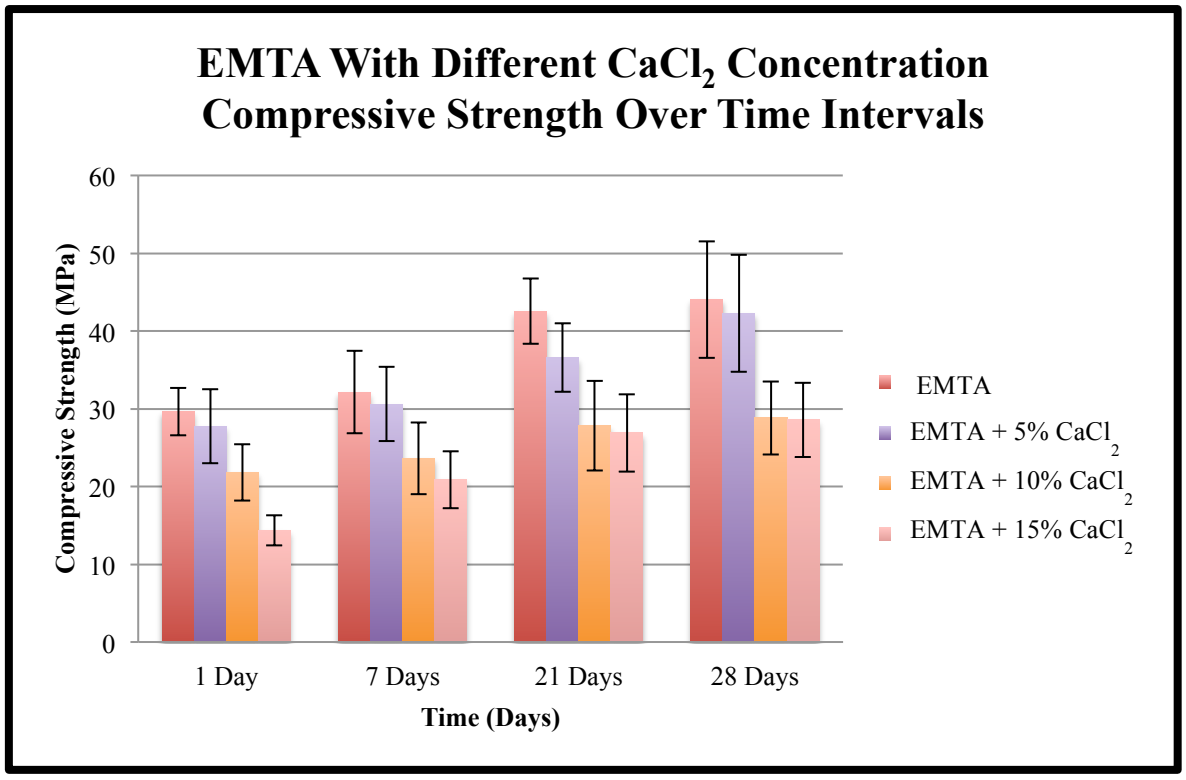


Figure 6-25: The compressive strength over time intervals of EMTA with different $CaCl_2$ concentrations.

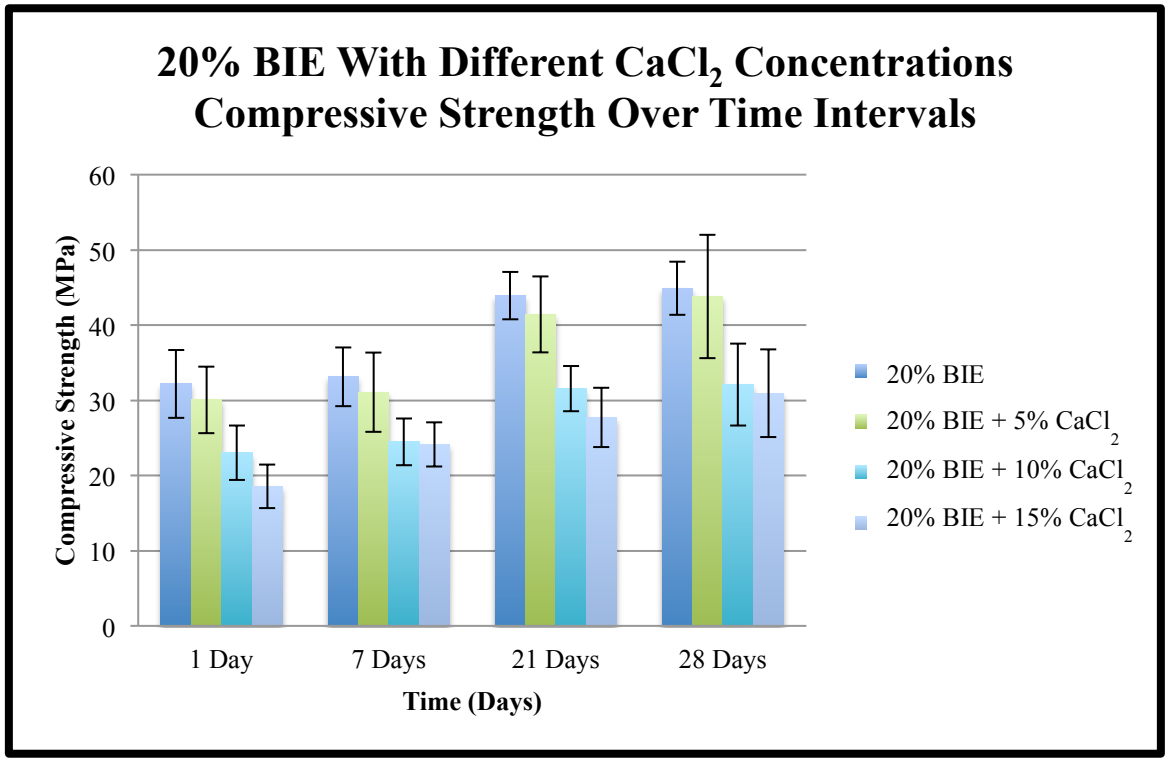


Figure 6-26: The compressive strength over time intervals of 20% BIE with different $CaCl_2$ concentrations.

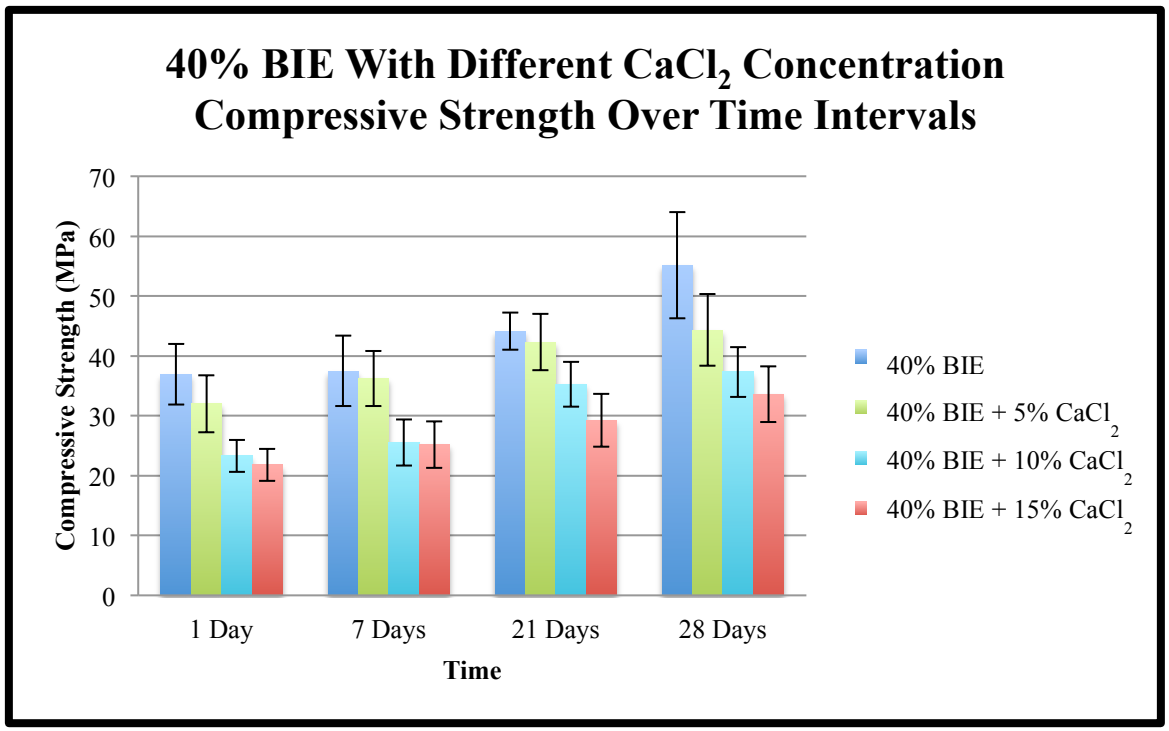


Figure 6-27: The compressive strength over time intervals of 40% BIE with different $CaCl_2$ concentrations.

Table 6-14: Two-Way ANOVA of Compressive Strength.

Variable	Type lll Sum of Squares	df	Mean Square	F-Value	p-Value
Material (Dycal®, EMTA, 20% BIE and 40% BIE)	21029.68	3	7009.89	332.42	< 0.0001
$CaCl_2$ Concentration	17134.95	3	5711.65	270.85	< 0.0001
Time	13275.87	3	4425.29	209.85	< 0.0001
Material * $CaCl_2$ Concentration * Time	1462.80	42	34.82	1.65	0.007

Table 6-15: Mean Compressive Strength and Results of Tukey Multiple Comparisons Test of All Materials at 1 Day.

Specimen (n=10)	Mean (MPa)	SD	Min	Max	Tukey's Letter (¥)	p < 0.05
Dycal® (1)	10.78	1.15	9.24	12.99	F	1& (2*,3*,4*,6*,7*,8*,9,10*,11*,12*, 13*)
EMTA (2)	29.62	3.03	23.65	33.70	B	2& (1*, 4, 5*,8, 9*,10,12,13)
EMTA + 5% $CaCl_2$ (3)	27.73	4.74	20.11	35.42	B C	3& (1*, 4, 5*,9*,10*,13)
EMTA + 10% $CaCl_2$ (4)	21.81	3.62	16.04	28.46	D	4& (1*, 2, 3, 5,6*,7, 10*,11*)
EMTA + 15% $CaCl_2$ (5)	14.36	1.93	11.26	17.13	E F	5& (2*,3*,4,6*,7*,8*,10*,11*,12*,13)
20% BIE (6)	32.16	4.76	24.73	40.02	A B	6& *(1,4,5,8,9,12,13)
20% BIE + 5% $CaCl_2$ (7)	31.05	4.42	23.80	35.53	B	7& (1*,4,5*,8,9*,10,12,13)
20% BIE + 10% $CaCl_2$ (8)	23.01	3.60	16.54	28.73	C D	8& (1*,2,5*,6*,7,10*,11*)
20% BIE + 15% $CaCl_2$ (9)	18.53	2.88	14.09	22.13	E D	9& (1,2*,3*,6*,7*,10*,11*)
40% BIE (10)	36.91	5.04	29.61	43.91	A	10& (1*,2,3*,4*,5*,7,8*,9*,12*,13*)
40% BIE + 5% $CaCl_2$ (11)	32.02	4.78	27.00	39.50	A B	11& *(1,4,5,8,9,12,13)
40% BIE + 10%$CaCl_2$ (12)	23.29	2.66	19.53	28.56	C D	12& (1*,2*,5*,6*,7,10*,11*)
40% BIE + 15% $CaCl_2$ (13)	21.80	2.68	17.62	26.75	D	13& (1*,2,3, 5,6*,7,10*,11*)

* $p < 0.0001$

(¥) Materials with the same letters were not statistically significant ($p > 0.05$).

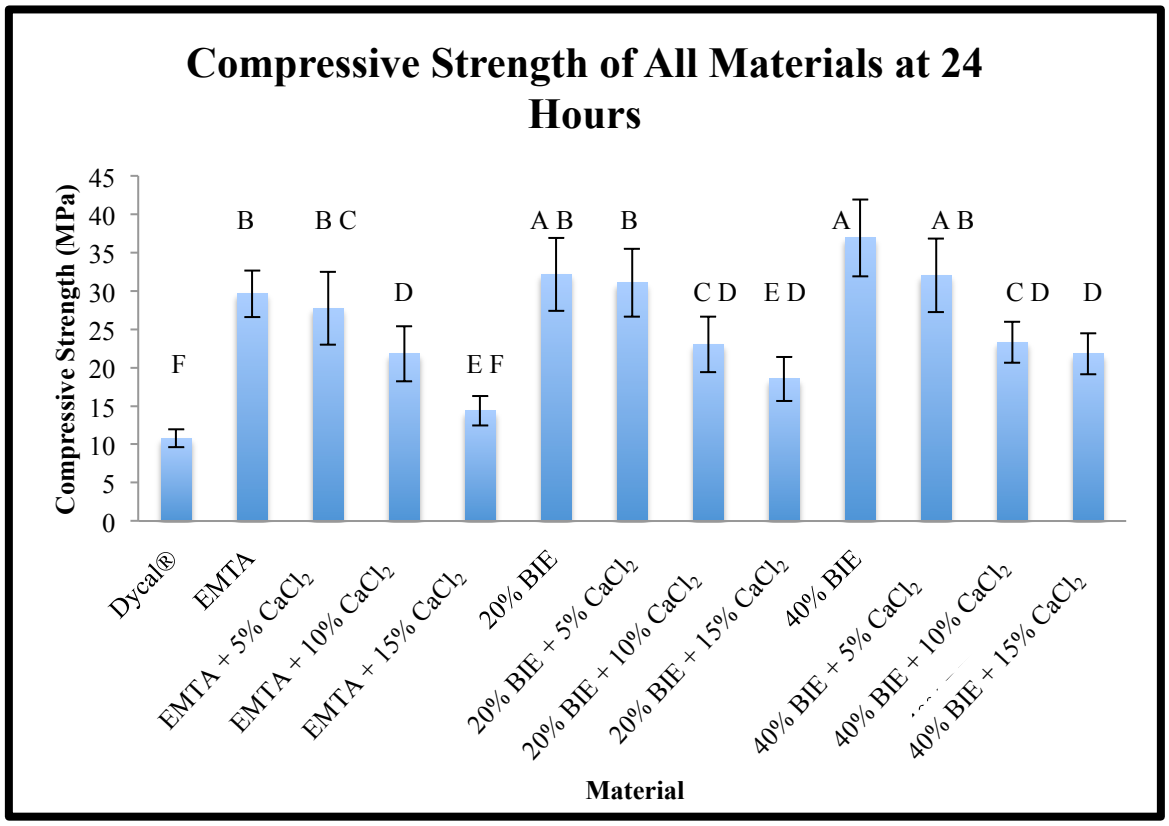


Figure 6-28: Compressive strength of all materials at 24 hours

Table 6-16: Mean Compressive Strength and Results of Tukey Multiple Comparisons Rest of All Materials at 7 Days.

Specimen (n=10)	Mean (MPa)	SD	Min	Max	Tukey's Letter (¥)	p-value <0.05
Dycal® (1)	12.06	2.04	9.33	15.90	F	1& (2*,3*,4*,5,6*,7*,8*,9*,10*, 11*,12*,13*)
EMTA (2)	32.14	5.29	21.80	38.18	A B	2& (1*,4,5*,8,9,12,13)
EMTA + 5% $CaCl_2$ (3)	30.58	5.04	21.27	35.26	B C D	3& (1*,4,5,10)
EMTA + 10% $CaCl_2$ (4)	23.62	4.62	17.73	28.55	E	4& (1*,2,3,6,7,10*,11*)
EMTA + 15% $CaCl_2$ (5)	20.86	3.65	15.62	25.57	E	5& (1,2*,3,6*,7*,10*,11*)
20% BIE (6)	33.13	3.91	21.18	37.82	A B	6& (1*,4,5*,8,9,12,13)
20% BIE + 5% $CaCl_2$ (7)	31.05	5.24	22.95	37.00	A B C	7& (1*,4,5*,8,9)
20% BIE + 10% $CaCl_2$ (8)	24.45	3.12	20.45	29.89	E D	8& (1*,2,6,7,10*,11*)
20% BIE + 15% $CaCl_2$ (9)	24.10	2.95	19.34	28.03	E D	9& (1*,2,6,7,10*,11*)
40% BIE (10)	37.46	5.88	27.98	45.40	A	10& (1*,3,4*,5*,8*,9*,12*,13*)
40% BIE + 5% $CaCl_2$ (11)	36.21	4.61	29.43	41.78	A B	11& *(1,4,5,8,9,12,13)
40% BIE + 10%$CaCl_2$ (12)	25.50	3.86	19.17	33.33	C D E	12& (1*,2,6,10*,11*)
40% BIE + 15% $CaCl_2$ (13)	25.18	3.88	20.67	31.21	C D E	13& (1*,2,6,10*,11*)

* $p < 0.0001$

(¥) Materials with the same letters were not statistically significant ($p > 0.05$)

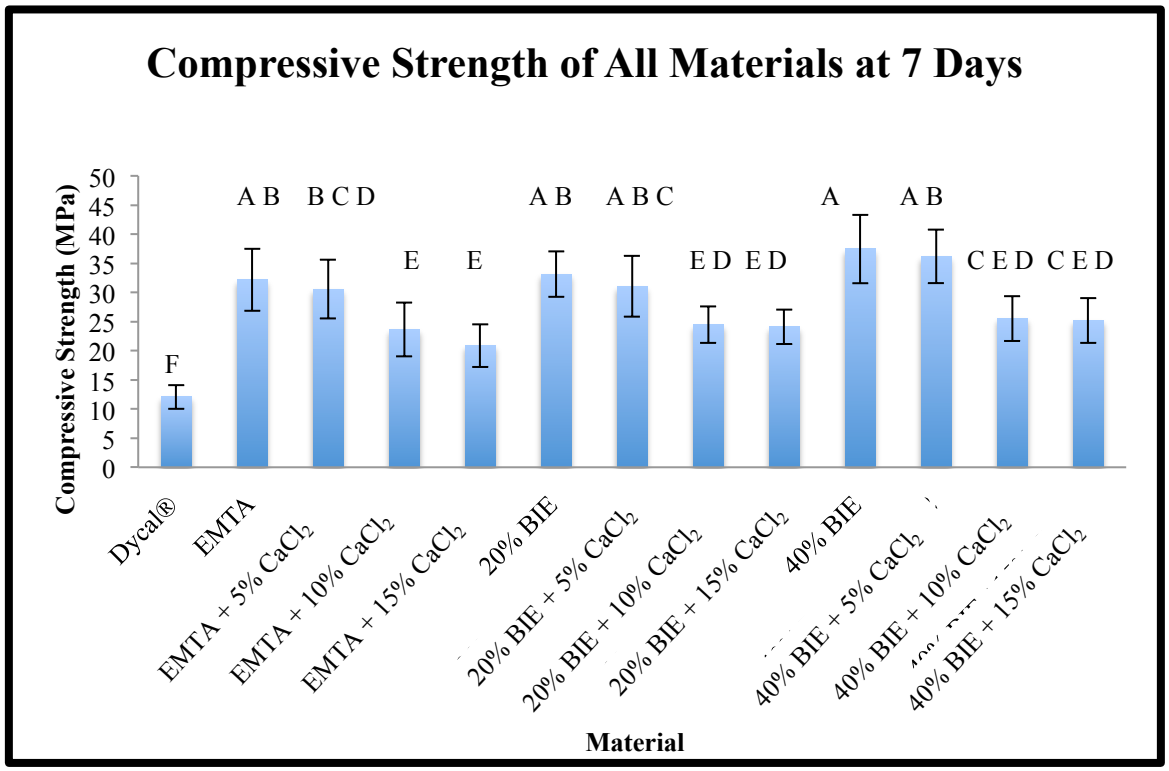


Figure 6-29: Compressive strength of all materials at 7 days

Table 6-17: Mean Compressive Strength and Results of Tukey Multiple Comparisons Rest of All Materials at 21 Days.

Specimen (n=10)	Mean (MPa)	SD	Min	Max	Tukey's Letter (¥)	p-value <0.05
Dycal® (1)	15.54	2.34	11.98	18.30	G	1& *(2,3,4,5,6,7,8,9,10,11,12,13)
EMTA (2)	42.52	4.19	35.17	47.70	A B	2& (1*,4*,5*,8*,9*,12,13*)
EMTA + 5% $CaCl_2$ (3)	36.56	4.38	28.46	42.00	B C D	3& (1*,4,5*,6,9,10,13)
EMTA + 10% $CaCl_2$ (4)	27.80	4.36	21.26	36.76	F	4& (1*,2*,3,6*,7*,10*,11*,12)
EMTA + 15% $CaCl_2$ (5)	26.88	4.96	19.91	34.19	F	5& (1*,2*,3*,6*,7*,10*,11*,12)
20% BIE (6)	43.91	3.17	38.78	49.51	A	6& (1*,3,4*,5*,8*,9*,12,13*)
20% BIE + 5% $CaCl_2$ (7)	41.41	5.53	34.56	50.50	A B C	7& *(1,4,5,8,9,13)
20% BIE + 10% $CaCl_2$ (8)	31.53	2.99	28.47	35.71	D E F	8& *(1,2,6,7,10,11)
20% BIE + 15% $CaCl_2$ (9)	27.73	3.96	21.78	32.59	F	9& (1*,2*,3,6*,7*,10*,11*,12)
40% BIE (10)	44.09	3.09	40.11	49.47	A	10& (1*,3,4*,5,*8*,9*,12,13*)
40% BIE + 5% $CaCl_2$ (11)	42.28	4.68	37.26	49.42	A B	11& (1*,4*,5*,8*,9*,12,13*)
40% BIE + 10%$CaCl_2$ (12)	35.27	3.76	29.35	40.95	C D E	12& (1*,2,4,5,6,9,10,11)
40% BIE + 15% $CaCl_2$ (13)	29.24	4.41	21.99	35.46	E F	13& (1*,2*,3,6*,7*,10*,11*)

* $p < 0.0001$

(¥) Materials with the same letters were not statistically significant ($p > 0.05$)

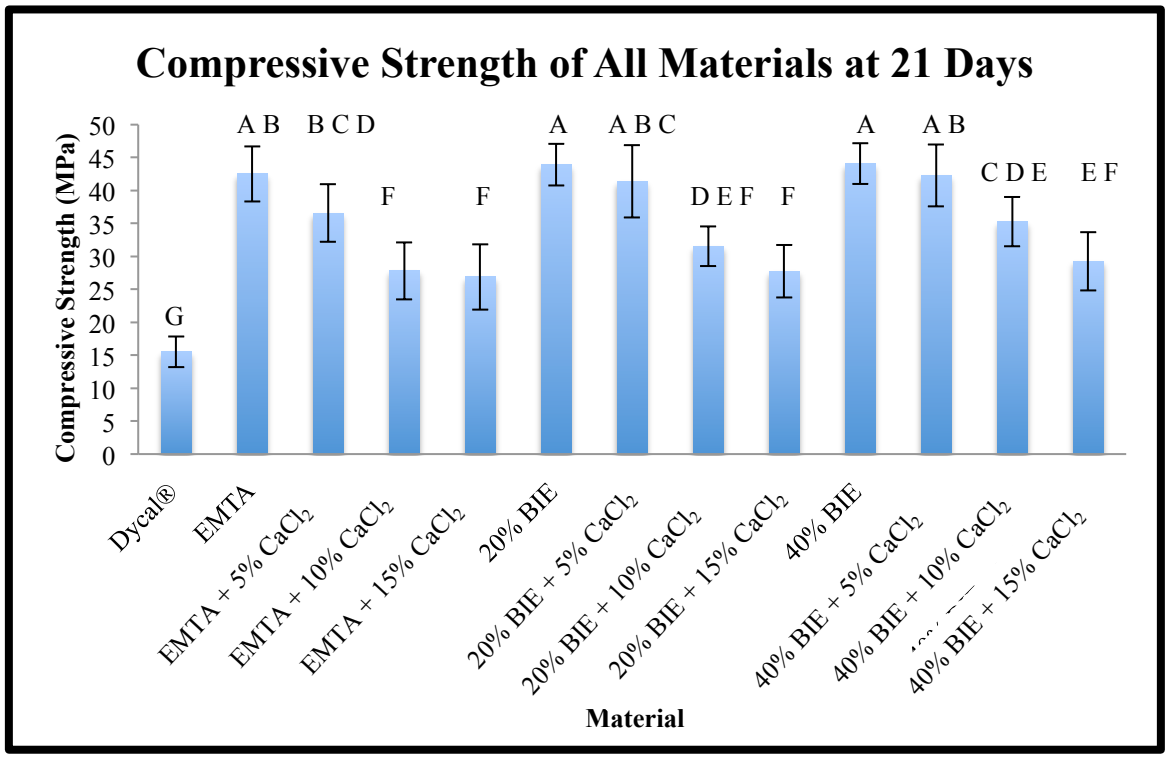


Figure 6-30: Compressive strength of all materials at 21 days

Table 6-18: Mean Compressive Strength and Results of Tukey Multiple Comparisons Rest of All Materials at 28 Days.

Specimen (n=10)	Mean (MPa)	SD	Min	Max	Tukey's Letter (¥)	p-value < 0.05
Dycal® (1)	19.76	3.32	15.14	24.11	E	1& (2*,3*,6*,7*,8,9,10*,11*,12*, 13*)
EMTA (2)	44.01	7.49	35.11	57.56	B	2& (1*,4*,5*,8,9,10,13)
EMTA + 5% $CaCl_2$ (3)	42.26	7.51	31.27	53.16	B C	3& (1*,4,5*,8,9,10)
EMTA + 10% $CaCl_2$ (4)	28.81	4.70	21.53	37.04	E D	4& (2*,3,6*,7*,10*,11*)
EMTA + 15% $CaCl_2$ (5)	28.55	4.76	21.18	36.21	E D	5& *(2,3,6,7,10,11)
20% BIE (6)	44.88	3.55	39.48	51.38	B	6& (1*,4*,5*,8,9*,10,13)
20% BIE + 5% $CaCl_2$ (7)	43.78	8.18	33.71	55.23	B	7& (1*,4*,5*,8,9,10,13)
20% BIE + 10% $CaCl_2$ (8)	32.09	5.45	24.21	39.98	D	8& (1,2,3,6,7,10*,11)
20% BIE + 15% $CaCl_2$ (9)	30.93	5.86	23.55	40.73	D	9& (1,2,3,6*,7,10*,11)
40% BIE (10)	55.13	8.85	41.87	67.83	A	10& (1*,2,3,4*,5*,6,7,8,9*,12*,13*)
40% BIE + 5% $CaCl_2$ (11)	44.31	5.99	30.62	49.70	B	11& (1*,4*,5*,8,9,13)
40% BIE + 10%$CaCl_2$ (12)	37.31	4.17	32.71	46.18	B C D	12& *(1,10)
40% BIE + 15% $CaCl_2$ (13)	33.57	4.66	26.58	41.03	C D	13& (1*,2,6,7,10*,11)

* $p < 0.0001$

(¥) Materials with the same letters were not statistically significant ($p > 0.05$)

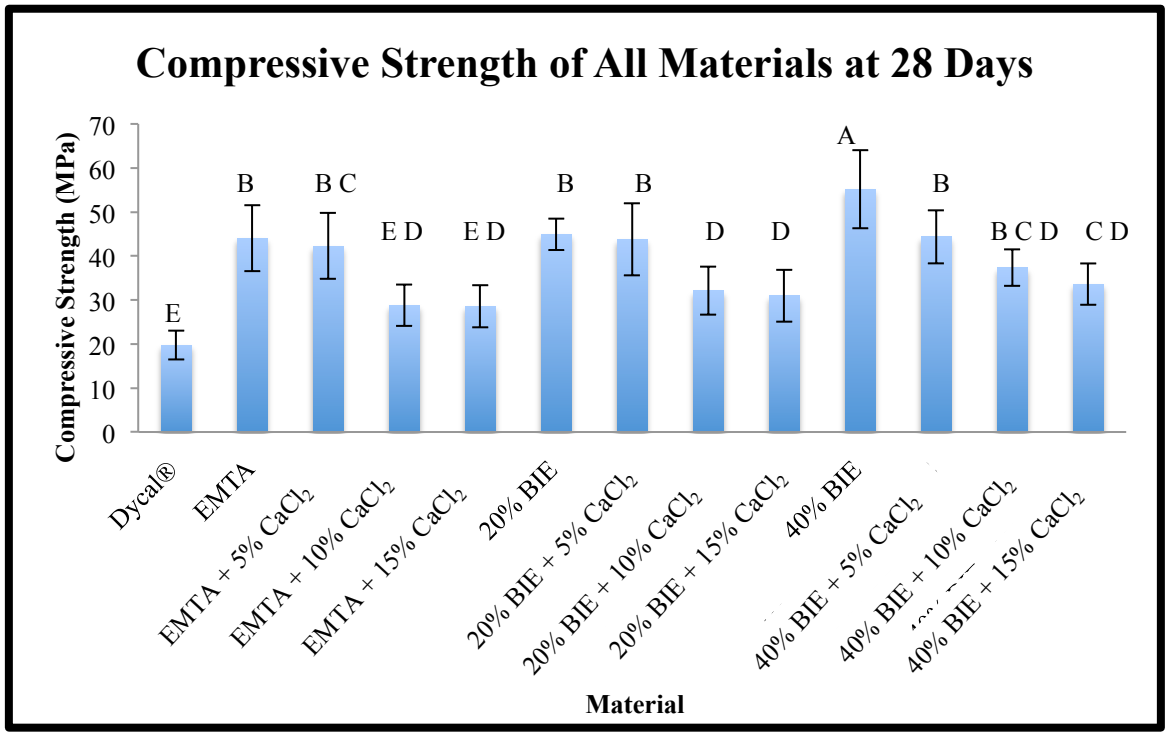


Figure 6-31: Compressive strength of all materials at 28 days

A linear regression model predicting the odds of having higher compressive strength based on $CaCl_2$ concentrations and time intervals was performed (Table 6-19). The effect of $CaCl_2$ and time accounted for 35% of the variability in the overall compressive strength (Adjusted $R^2 = 0.35$, $p < 0.0001$).

This model shows that each unit (5% increments) increase in the concentration of $CaCl_2$ resulted in a decrease of the compressive strength by 3.144 MPa, and each increase in the time interval resulted in an increase of the compressive strength by 4.449 MPa after controlling for other variables (material type, solubility, and pH).

Table 6-19: Linear Regression Model Predicting Higher Compressive Strength.

Variable	Estimate	p-value
$CaCl_2$ concentration	-3.144	< 0.0001
Time	4.449	< 0.0001

6.5. Solubility

Table 6-20 represents the means of the initial weight of all tested materials, weight change at different time intervals, and the mean percent solubility compared to initial weight of all groups at days 1, 7, 21, and 28. The table shows that Dycal® was the most soluble material during all time intervals, and solubility increased over time and by adding $CaCl_2$.

Table 6-20: Means of Initial Weight, Weight Loss, and Solubility of All Groups at 1,7,21, and 28 Days.

Material	Initial weight (g)	Weight 1 day (g)	Solubility at 1 day %	Weight 7 days (g)	Solubility at 7 day %	Weight 21 days (g)	Solubility at 21 days %	Weight 28 days (g)	Solubility at 28 days %
Dycal®	1.9685	1.8005	8.5472	1.6218	17.6756	1.4799	24.9048	1.3652	30.7683
EMTA	1.7917	1.7819	0.5527	1.7756	0.9127	1.7699	1.2298	1.7652	1.4953
EMTA +5% $CaCl_2$	1.6230	1.5773	2.8152	1.5370	5.30389	1.5084	7.0505	1.4891	8.2348
EMTA +10% $CaCl_2$	2.0628	1.8998	7.8968	1.7719	14.1025	1.6680	19.1696	1.6199	21.5144
EMTA +15% $CaCl_2$	2.1614	1.9817	8.3201	1.8357	15.1186	1.6915	21.8093	1.6177	25.2943
20%BIE	1.7310	1.7222	0.5326	1.7149	0.96489	1.7079	1.4064	1.7019	1.7546
20%BIE + 5% $CaCl_2$	1.9638	1.8337	6.6356	1.7906	8.86577	1.7515	10.8657	1.7250	12.2067
20%BIE + 10% $CaCl_2$	2.1614	1.9746	8.5777	1.8587	13.9813	1.7447	19.3008	1.6906	21.8173
20%BIE + 15% $CaCl_2$	2.0850	1.9075	8.5202	1.7868	14.3598	1.7129	17.8938	1.6449	21.1603
40% BIE	1.6510	1.6460	0.2913	1.6422	0.51537	1.6386	0.7544	1.6357	0.9399
40%BIE + 5% $CaCl_2$.	1.6758	1.5732	5.8534	1.5483	7.3817	1.5284	8.6000	1.5013	10.2437
40%BIE + 10% $CaCl_2$.	1.9215	1.7755	7.6017	1.6859	12.2545	1.6144	15.9595	1.5748	18.0656
40%BIE + 15% $CaCl_2$	2.1665	2.0191	6.8171	1.9031	12.2926	1.8215	16.1167	1.7736	18.2673

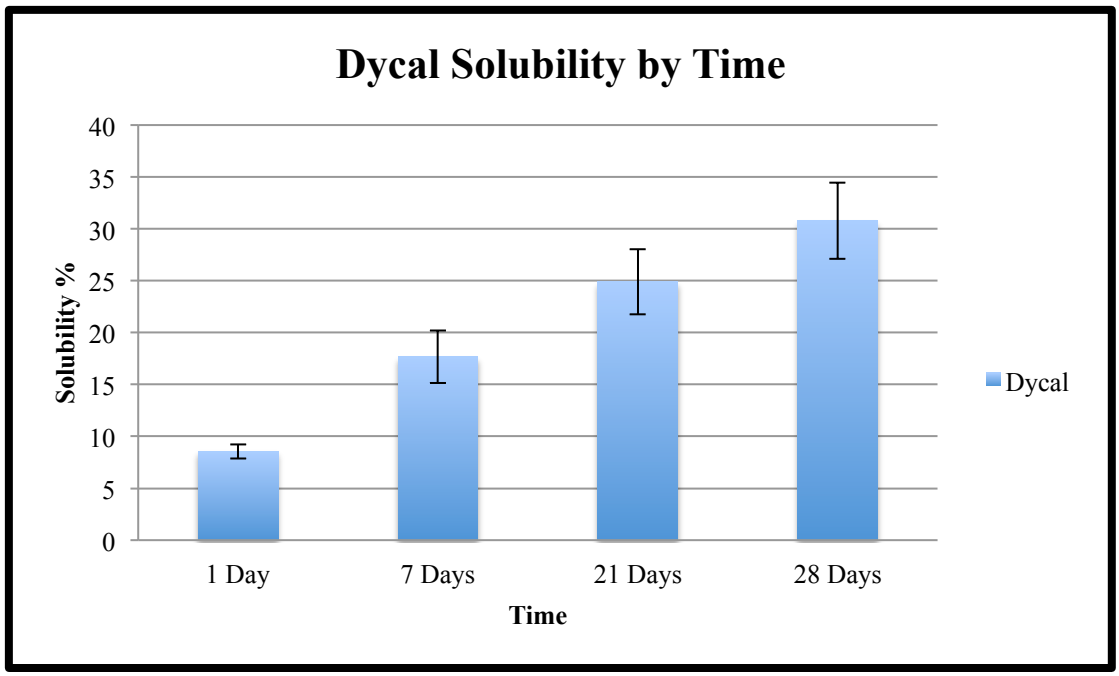


Figure 6-32: Dycal® solubility by time intervals.

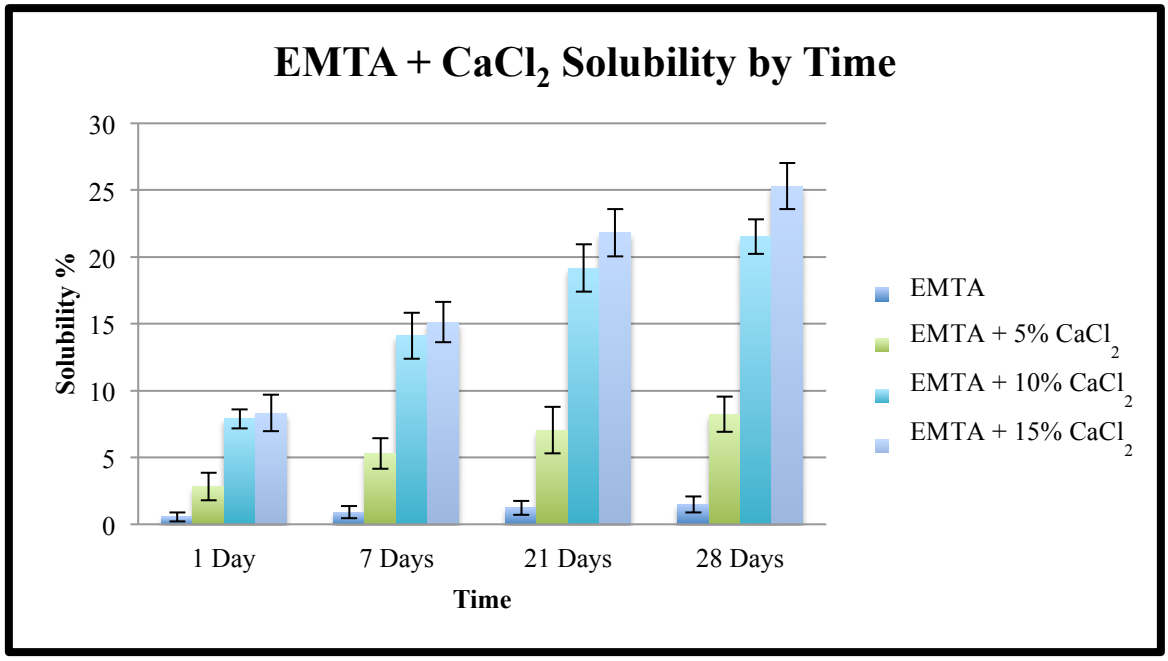


Figure 6-33: EMTA solubility with different $CaCl_2$ concentrations by time intervals.

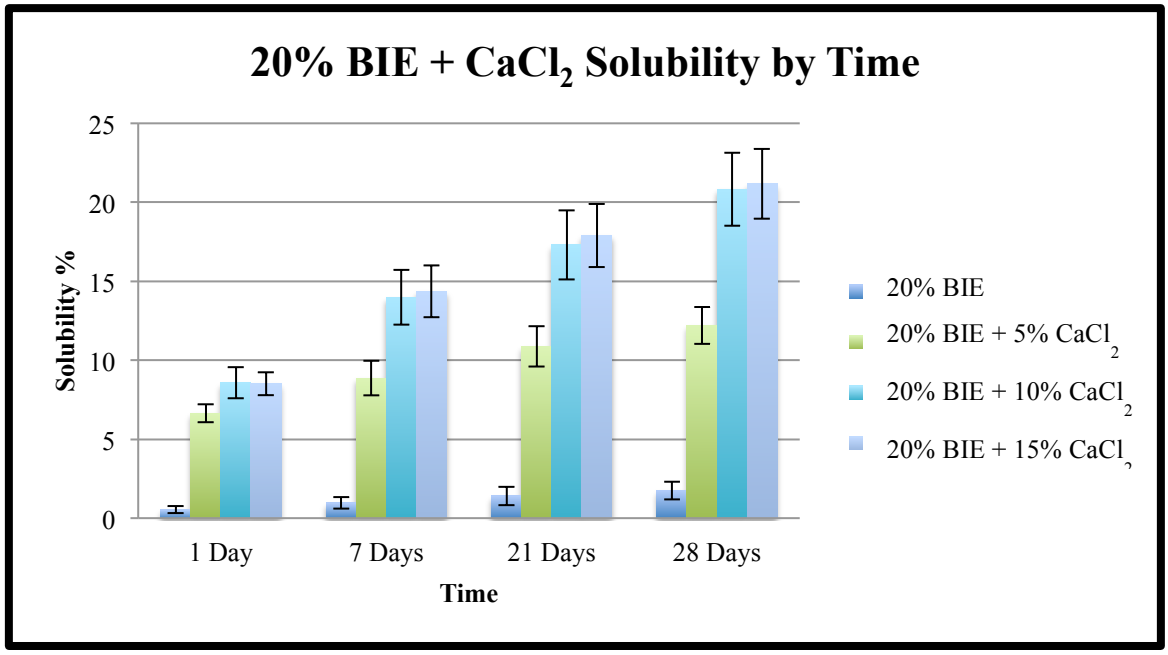


Figure 6-34: 20% BIE solubility with different $CaCl_2$ concentrations by time intervals.

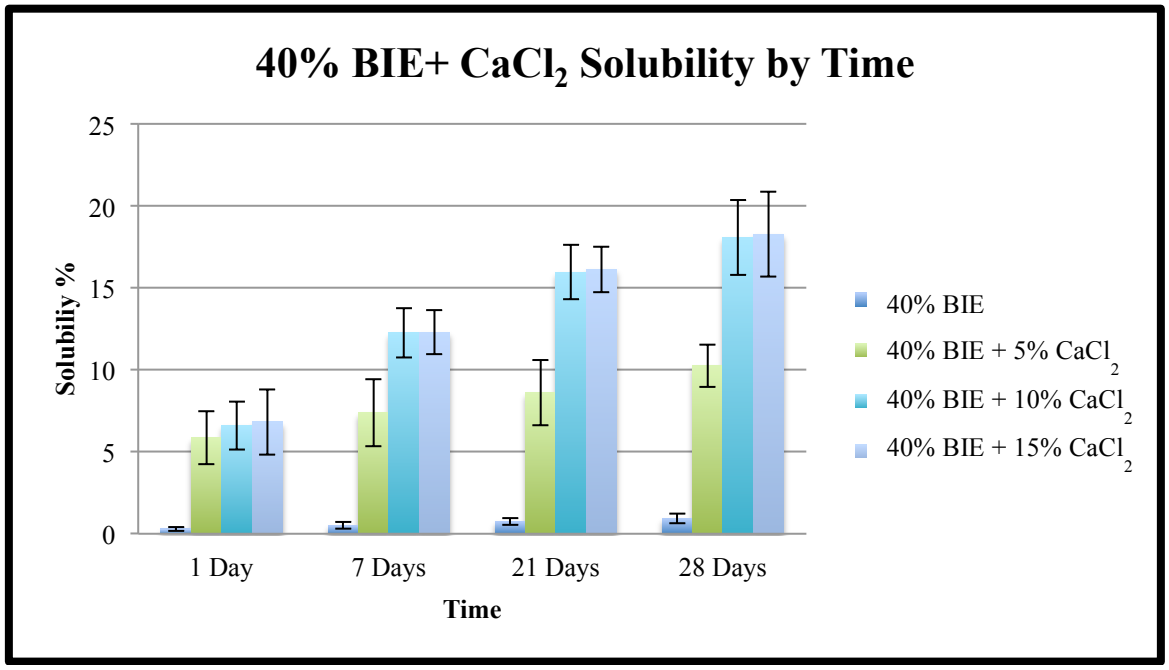


Figure 6-35: 40% BIE solubility with different $CaCl_2$ concentrations by Time.

The results of the two-way ANOVA (Table 6- 21) showed that solubility was statistically significant different among the groups ($p < 0.0001$). The solubility was statistically affected by the type of material (Dycal®, EMTA, 20% BIE, and 40% BIE), the different concentrations of $CaCl_2$, and the different time intervals. An interaction was also found between the three variables (material, $CaCl_2$ concentrations, and time).

The mean solubility of all tested materials, the statistical analysis of two-way ANOVA at $p < 0.05$, and Tukey's multiple comparison test are presented at each time interval in Tables 6-22 to 6-25. A graphical presentation of all materials solubility vs. each time interval is presented in Figures 6-36 to 6-39.

At all time intervals, the highest solubility was seen with Dycal®. The lowest solubility was seen with 40% BIE. This difference was statistically significant ($p < 0.0001$). EMTA, 20% BIE, and 40% BIE materials had comparable levels of solubility ($p > 0.05$) during all time intervals.

Adding $CaCl_2$ to any material significantly increased solubility in comparison to the same material without $CaCl_2$ ($p < 0.0001$). However, there was no significant difference in solubility between adding 10% and 15% $CaCl_2$ ($p > 0.05$).

Table 6-21: Two-Way ANOVA of Solubility

Variable	Type lll Sum of Squares	df	Mean Square	F-Value	p-Value
Material (Dycal®, EMTA, 20% BIE and 40% BIE)	11678.59	3	3892.86	622.75	< 0.0001
$CaCl_2$ Concentration	17034.52	3	5678.17	908.36	< 0.0001
Time	6196.73	3	2065.57	330.44	< 0.0001
Material * $CaCl_2$ Concentration * Time	3679.77	42	87.61	14.02	< 0.0001

Table 6-22: Mean Solubility and Results of Tukey Multiple Comparisons Test of All Materials at 24 Hours.

Specimen (n=10)	Mean %	SD	Min	Max	Tukey's Letter (¥)	p-value < 0.05
Dycal® (1)	8.54	0.67	7.21	9.48	A	1& *(2,6,10)
EMTA (2)	0.55	0.31	0.16	1.17	B	2& *(1,4,5,7,8,9,11,12,13)
EMTA + 5% $CaCl_2$ (3)	6.81	0.54	0.97	4.60	A	3& *(2,6,10)
EMTA + 10% $CaCl_2$ (4)	7.89	0.72	6.07	8.54	A	4& *(2,6,10)
EMTA + 15% $CaCl_2$ (5)	8.32	1.36	5.55	9.66	A	5& *(2,6,10)
20% BIE (6)	0.53	0.22	0.25	1.08	B	6& *(1,4,5,7,8,9,11,12,13)
20% BIE + 5% $CaCl_2$ (7)	6.63	0.56	5.79	7.56	A	7& (2*, 6*,10*)
20% BIE + 10% $CaCl_2$ (8)	8.57	0.98	4.98	11.80	A	8& *(2,6,10)
20% BIE + 15% $CaCl_2$ (9)	8.52	0.73	7.50	9.58	A	9& *(2,6,10)
40% BIE (10)	0.29	0.12	0.08	0.46	B	10& *(1,3,4,5,7,8,9,11,12,13)
40% BIE + 5% $CaCl_2$ (11)	5.85	0.6	0.31	18.51	A	11& (2*,6*,10*)
40% BIE + 10%$CaCl_2$ (12)	6.6	1.5	4.15	10.14	A	12& *(2,6,10)
40% BIE + 15% $CaCl_2$ (13)	6.81	1.46	5.29	10.13	A	13& (2*,6*,10*)

* $p < 0.0001$

(¥) Materials with the same letters were not statistically significant ($p > 0.05$)

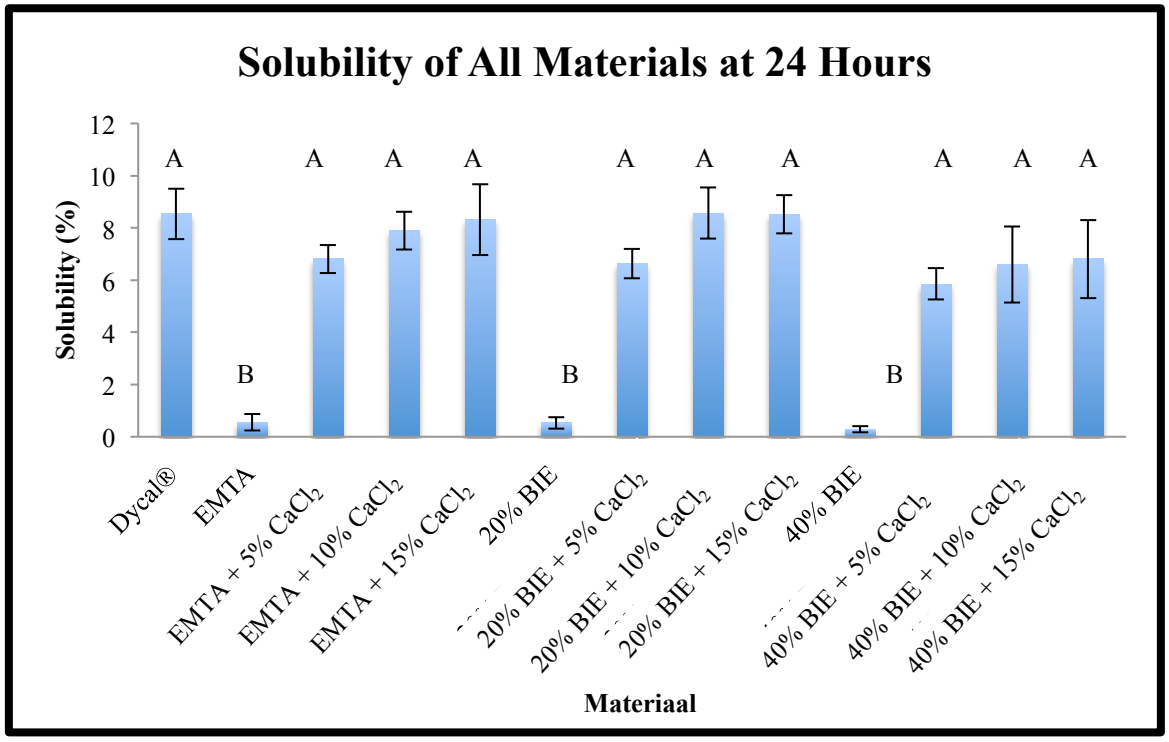


Figure 6-36: Solubility of all materials at 24 hours.

Table 6-23: Mean Solubility and Results of Tukey Multiple Comparisons Test of All Materials at 7 Days.

Specimen (n=10)	Mean %	SD	Min	Max	Tukey's Letter (¥)	p-value < 0.05
Dycal® (1)	17.67	1.53	14.29	21.98	A	1& (2*,3*,4,6*,7*,10*,11*,12*, 13*)
EMTA (2)	0.91	0.20	0.22	1.77	F	2& (1*,3,4*,5*,7*,8*,9*,11*,12*, 13*)
EMTA + 5% $CaCl_2$ (3)	5.30	1.14	1.80	10.00	E	3& (1*,2,4*,5*,6,8*,9*,10,12*, 13*)
EMTA + 10% $CaCl_2$ (4)	14.12	1.71	10.68	16.11	B	4& (1,2*,3*,6*,7*,10*,11*)
EMTA + 15% $CaCl_2$ (5)	15.11	1.50	12.95	17.89	A B	5& *(2,3,6,7,10,11)
20% BIE (6)	0.96	0.18	0.40	1.72	F	6& (1*,3,4*,5*,7*,8*,9*,11*,12*, 13*)
20% BIE + 5% $CaCl_2$ (7)	8.86	1.10	7.56	10.61	C D E	7& (1*,2*,4,5*,6*,8,9*,10*)
20% BIE + 10% $CaCl_2$ (8)	13.98	1.72	10.28	18.29	A B	8& (2*,3*,6*,7,10*,11*)
20% BIE + 15% $CaCl_2$ (9)	14.35	1.63	11.86	16.70	A B	9& *(2,3,6,7,10,11)
40% BIE (10)	0.51	0.2	0.17	0.80	F	10& (1*,3,4*,5*,7*,8*,9*,11*,12*, 13*)
40% BIE + 5% $CaCl_2$ (11)	7.38	1.41	1.32	19.02	D E	11& (1*,2*,4*,5*,6*,8*,9*,10*,12, 13)
40% BIE + 10%$CaCl_2$ (12)	12.25	1.5	9.88	14.42	B C	12& (1*,2*,3*,5*,6*,10*,11)
40% BIE + 15% $CaCl_2$ (13)	12.29	2.33	8.41	15.55	B C	13& (1*,2*,3*,6*,10*,11)

* $p < 0.0001$

(¥) Materials with the same letters were not statistically significant ($p > 0.05$)

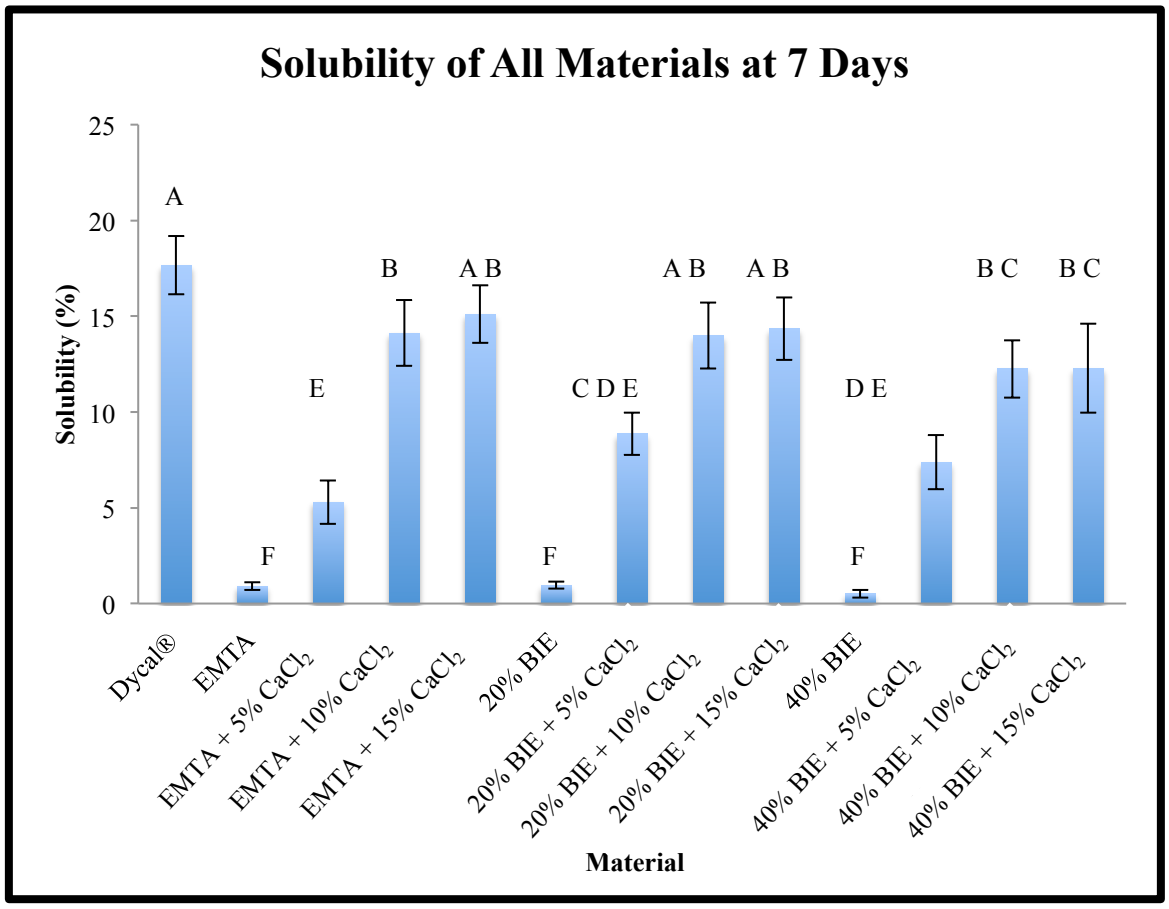


Figure 6-37: Solubility of all materials at 7 days.

Table 6-24: Mean Solubility and Results of Tukey Multiple Comparisons Test of All Materials at 21 Days

Specimen (n=10)	Mean %	SD	Min	Max	Tukey's Letter (¥)	p-value < 0.05
Dycal® (1)	24.90	2.14	19.16	29.99	A	1& (2*,3*,4,6*,7*,8,9*,10*,11*, 12*,13*)
EMTA (2)	1.22	0.52	0.39	2.22	E	2& (1*,3,4*,5*,7*,8*,9*,11*,12*, 13*)
EMTA + 5% $CaCl_2$ (3)	7.05	1.74	2.43	12.60	D	3& (1*,2,4*,5*,6,8*,9*,10*,12*, 13*)
EMTA + 10% $CaCl_2$ (4)	19.16	1.77	14.65	25.31	B C	4& (1,2*,3*,6*,7*,10*,11*)
EMTA + 15% $CaCl_2$ (5)	21.80	1.76	19.14	24.78	A B	5& (2*,3*,6*,7*,10*,11*,12,13)
20% BIE (6)	1.40	0.58	0.63	2.719	E	6& (1*,3,4*,5*,7*,8*,9*,11*,12*, 13*)
20% BIE + 5% $CaCl_2$ (7)	10.86	1.27	9.21	12.74	D	7& (1*,2*,4*,5*,6*,8*,9*,10*,12, 13)
20% BIE + 10% $CaCl_2$ (8)	17.30	2.18	14.70	23.57	B C	8& (1,2*,3*,6*,7*,10*,11*)
20% BIE + 15% $CaCl_2$ (9)	17.89	1.99	15.91	21.13	B C	9& *(1,2,3,6,7,10,11)
40% BIE (10)	0.75	0.21	0.46	1.11	E	10& *(1,3,4,5,7,8,9,11,12,13)
40% BIE + 5% $CaCl_2$ (11)	8.60	2.98	2.22	19.57	D	11& *(1,2,4,5,6,8,9,10,12,13)
40% BIE + 10%$CaCl_2$ (12)	15.95	1.65	12.51	18.22	C	12& (1*,2*,3*,5,6*,7,10*,11*)
40% BIE + 15% $CaCl_2$ (13)	16.11	2.38	11.65	19.03	C	13& (1*,2*,3*,5,6*,7,10*,11*)

* $p < 0.0001$

(¥) Materials with the same letters were not statistically significant ($p > 0.05$)

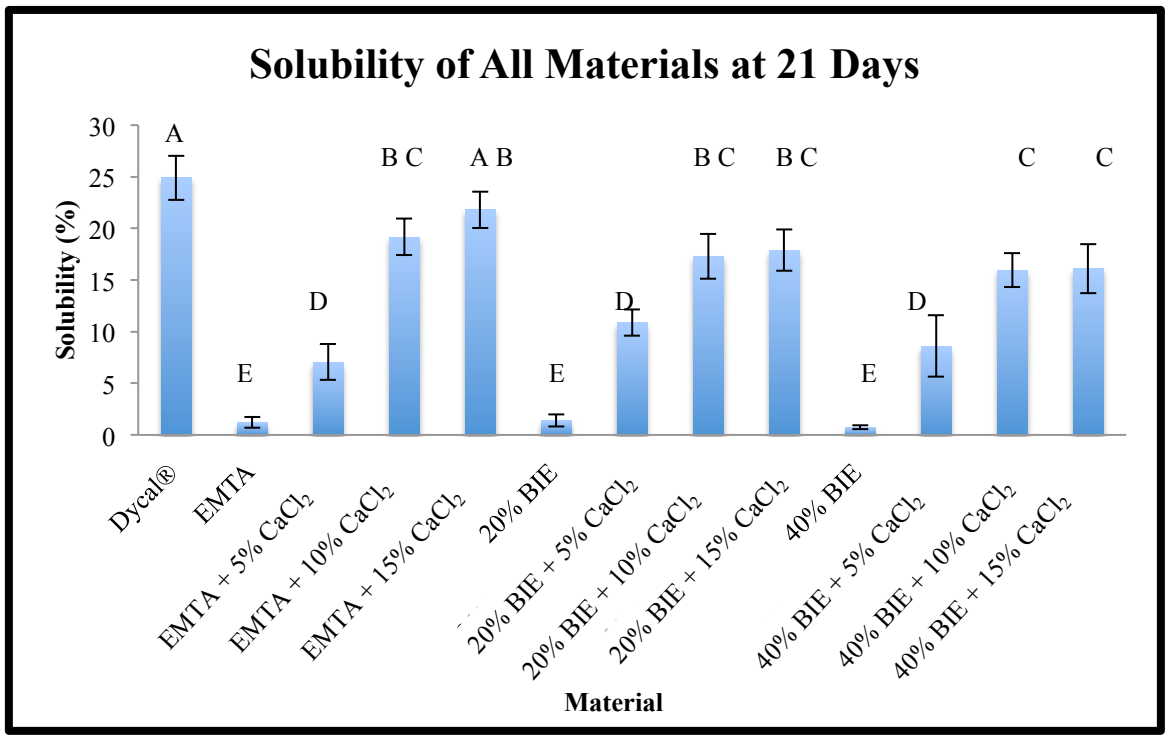


Figure 6-38: Solubility of all materials at 21 days.

Table 6-25: Mean Solubility and Results of Tukey Multiple Comparisons Test of All Materials at 28 Days

Specimen (n=10)	Mean %	SD	Min	Max	Tukey's Letter (¥)	p-value
Dycal® (1)	30.76	1.68	24.31	37.91	A	1& (2*,3*,4*,5,6*,7*,8*,9*,10*, 11*,12*,13*)
EMTA (2)	1.75	0.61	0.55	2.73	E	2& *(1,3,4,5,7,8,9,11,12,13)
EMTA + 5% $CaCl_2$ (3)	8.23	2.31	2.50	14.33	D	3& (1*,2*,4*,5*,6,8*,9*,10*,12*,13*)
EMTA + 10% $CaCl_2$ (4)	21.51	2.29	16.81	27.41	B C	4& *(1,2,3,6,7,10,11)
EMTA + 15% $CaCl_2$ (5)	25.29	1.73	20.55	29.46	B	5& (1,2*,3*,6*,7*,10*,11*,12*,13*)
20% BIE (6)	1.49	0.35	1.15	3.26	E	6& *(1,2,3,5,7,8,9,10,11)
20% BIE + 5% $CaCl_2$ (7)	12.20	2.17	10.59	14.20	D	7& (1*,2*,4*,5*,6*,8*,9*,10*,12,13)
20% BIE + 10% $CaCl_2$ (8)	21.81	2.3	16.67	26.15	B C	8& *(1,2,3,6,7,10,11)
20% BIE + 15% $CaCl_2$ (9)	21.16	2.21	17.70	24.73	B C	9& *(1,2,3,6,7,10,11)
40% BIE (10)	0.93	0.29	0.636	1.45	E	10& *(1,3,4,5,7,8,9,11,12,13)
40% BIE + 5% $CaCl_2$ (11)	10.24	2.29	2.73	20.15	D	11& *(1,2,4,5,6,8,9,10,12,13)
40% BIE + 10%$CaCl_2$ (12)	18.06	1.29	14.95	21.75	C	12& (1*,2*,3*,5*,6*,7,10*,11*)
40% BIE + 15% $CaCl_2$ (13)	18.26	1.58	13.39	20.87	C	13& (1*,2*,3*,5*,6*,7,10*,11*)

* $p < 0.0001$

(¥) Materials with the same letters were not statistically significant ($p > 0.05$)

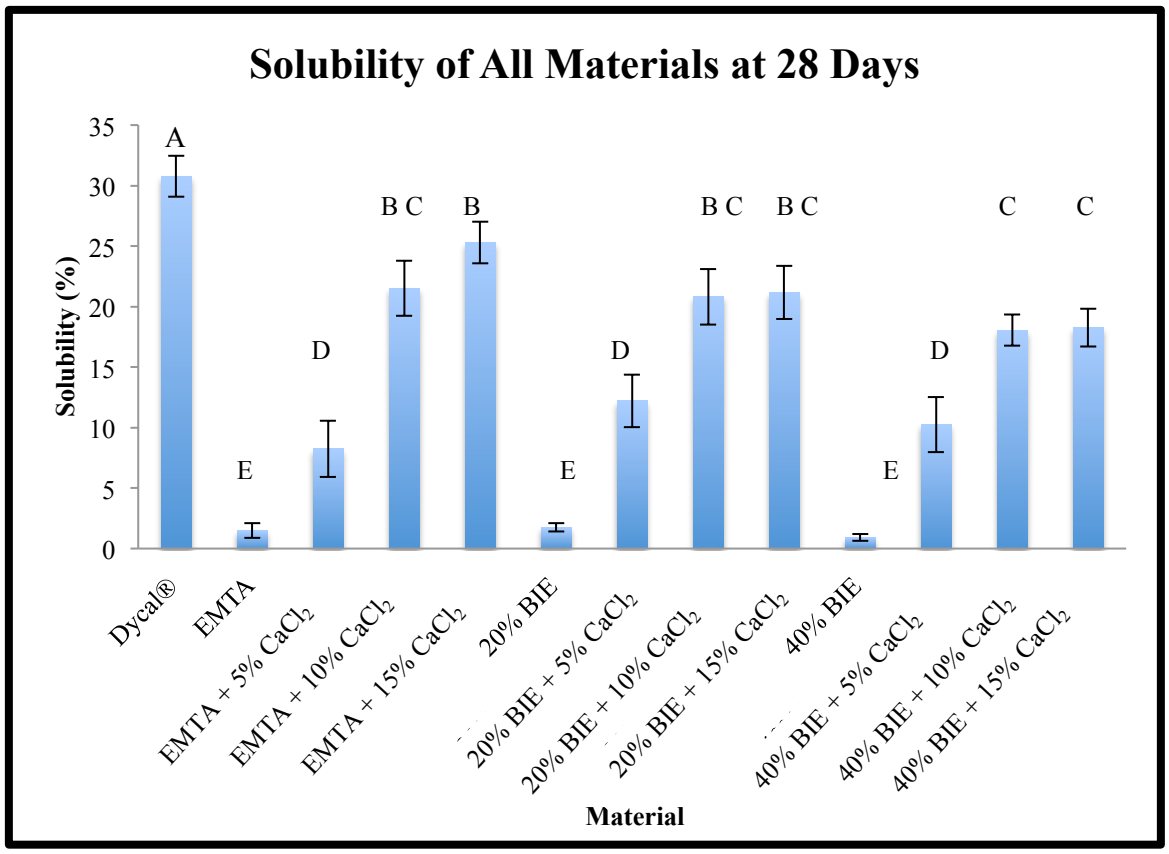


Figure 6-39: Solubility of All Materials at 28 Days.

A linear regression model predicting the odds of having higher solubility based on $CaCl_2$ concentration and time intervals was performed (Table 6-26). The effect of $CaCl_2$ and time accounted for 43% of the variability in the solubility of the material (Adjusted $R^2 = 043$, $p < 0.0001$).

This model shows that each 5% increments increase in the concentration of $CaCl_2$, will resulted in an increase in the solubility of 3.602%, and each increase in time interval resulted in increase in the solubility of 3.059%, after controlling for other variables.

Table 6-26: Linear Regression Model Predicting Higher Solubility Levels.

Variable	Estimate	p-value
$CaCl_2$ concentration	3.602	<0.0001
Time	3.059	<0.0001

DISCUSSION

7. DISCUSSION

The ideal pulp capping material requires the formation of a continuous, reparative dentin bridge. In addition, such qualities as fast setting time and good physical and mechanical properties are desirable so that the treatment may be completed in one visit and allow enough time for tissue healing. Calcium hydroxide dental materials has been used for many years in pulp capping due to its fast setting time and its ability to induce the formation of a dental barrier to protect the pulp. However, after its application, it often becomes a poorly formed calcified bridge instead of a sound dentin structure. The material has low physical and mechanical properties, including a high pH level, high solubility, and low compressive strength. For those reasons, scientists have started the investigations to develop a new pulp capping material with better physical and mechanical properties that is capable of forming dentin bridge. The use of MTA material in pulp capping began in the 1990s, and the literature has found that MTA has superior mechanical and physical properties compared to calcium hydroxide. However, MTA still lacks the ability to form a good dentin bridge, and the long setting time makes it inconvenient for use in dental clinic. For those reasons, and after the discovery of BIE material by Dr. Chou that has showed excellent biocompatibility results and the ability to induce dentin formation, we started the investigation of the material's mechanical and physical properties of a novel pulp capping material that contains BIE.

7.1. Particle Size and Mechanical Properties

The correlation between particle size of the material and its physical properties has been well documented in the literature (Dammarchke et al., 2005; Frigioine, 1976; Osbaeck, 1989). Studies have shown that materials with smaller, finer particles size are stronger, are less porous, exhibit less shrinkage, and a have lower propensity to cracking than other materials with larger particle size (Bentz et al., 1999; Shane et al., 1999).

In this study, we controlled the effect of particle size on the physical properties of different materials by ensuring that all materials had a particle size within the same range of less than 10 μm. This range was chosen based on the particle size of the control group (Dycal®). Except for the BIE material, the components of our groups showed particle sizes of less than 10 μm. BIE showed the largest particle size, ranging from 275 μm to 682 μm. Thus, the BIE material was subjected to grinding and sieves to create particles less than 10 μm.

7.2. Setting Time

The results of this study showed that without adding $CaCl_2$ Dycal® had the shortest setting time of 5.80 ± 1.13 minutes ($p < 0.0001$), followed by 40% BIE (60.00 ± 6.66 minutes), and 20% BIE (67.03 ± 13.34 minutes). EMTA had the longest setting time of 114.00 ± 10.74 minutes ($p<0.0001$). Adding 10% and 15% of $CaCl_2$ to 40% BIE decreased the material setting time to 14.0 ± 2.1 minutes, and 9.40 ± 0.96 minutes respectively, which was comparable to setting time of Dycal® ($p > 0.05$).

The main difference between the EMTA and the BIE containing materials (both 20% and 40% BIE), is the presence of BIE material which has high concentration of silica, and the absence of gypsum (calcium sulfate) in the composition of 20% and 40% BIE. Gypsum constitutes 5% of EMTA material and it is added to retard the material's setting time. Camilleri et al., in 2006 found that removal of gypsum material from MTA results in flash setting. The difference between the 20% BIE and 40% BIE is the concentration of the BIE material (mainly composed of silicon, calcium, and phosphorous), and the concentration of Portland cement. The 40% BIE material had more BIE material and less Portland cement.

The effects of adding SiO_2 to MTA and Portland cement material have been studied. Results showed that the SiO_2 behaved as a filler to improve the microstructure of the material and accelerate the hydration process. The silica particles acted as nuclei during the hydration process, and because of its high surface energy, it allowed the generation of more nucleation sites for the formation of products' hydration (Akbari et al., 2013).

In 2013, Akbari et al. compared the setting time of MTA and MTA with two different concentrations (8% and 10%) of nano-SiO_2 size 13–16 nm. In this study, the materials were placed in a cylindrical stainless steel mold (10 mm in diameter and 5 mm in height), and a 1.0-mm-diameter flat-end indenter was used with a 400 g load and carefully lowered vertically to the surface of the tested material. This procedure was repeated every 60 seconds, and the final setting time was recorded when the needle failed to make an indent in the material. The results showed the setting time when adding 8% or 10% of SiO_2 (202.33 ± 0.31 and 199.33 ± 0.31 minutes, respectively) was statistically shorter than the setting time of MTA (229.66 ± 0.31 minutes; $p = 0.003$). There was no statistically significant difference between the two SiO_2 groups ($p > 0.05$).

Our study is in agreement with Akbari's study, where the setting time of EMAT (114.00 ± 10.74 minutes) was statistically longer than silica-containing materials 20% BIE with setting time of 67.00 ± 13.37 minutes and 40% BIE 60.00 ± 6.66 minutes at $p < 0.0001$.

Although MTA material has acceptable mechanical properties, one of its major disadvantages is the long setting time, which frequently requires a second treatment appointment. In addition, the long setting time may result in the cement being displaced due to manipulation or irrigation. Therefore decreasing the setting time of MTA to one that is possible to accomplish in a single dental visit would be greatly favorable.

In this study, the setting times of different study materials with and without $CaCl_2$ were investigated. The setting time was determined according to a method similar to the

methods described by the International Organization of Standardization (ISO) 6876 (2002) for dental root canal sealing materials. Torabinejad et al. modified this technique and only measured the final setting time of the of MTA material, instead of measuring the initial and final setting time (Torabinejad et al., 1995 (c)). In addition, other studies followed the same technique concept to investigate the setting time of MTA (Islam & Yap, 2006; Monts, 2004; Spencer, 2004).

Abdullah et al. (2002) reported that the setting time of Portland cement was successfully reduced by the addition of 10% and 15% calcium chloride without affecting the material's biocompatibility. According to Bortoluzzi et al. $CaCl_2$ penetrated the pores of cements and accelerated the hydration reaction of silicates, which reduced their crystallization time, thus hastening the final setting time of the material (Bortoluzzi et al., 2009)

The effect of $CaCl_2$ in decreasing the setting time of Portland cement and MTA has also studied previously. In Bortoluzzi et al. study in 2009, the results showed a significant reduction in the setting time of WMTA after adding 10% $CaCl_2$ from 48 ± 0.87 minutes to 31 ± 2.0 minutes ($p < 0.05$). In Lee et al. study, the results showed a reduction in the setting time of MTA after adding 10% $CaCl_2$ from 108.1 ± 1.6 minutes to 74.0 ± 0.6 minutes ($p < 0.01$) (Lee et. al., 2011). In 2015, Prasad et al. conducted a comparative study about setting time, pH level, and compressive strength between EMTA and different additive including 10% $CaCl_2$. The results showed a statistical significant difference in setting time between MTA

mixed with distilled water (133.10 ± 7.84 minutes) and MTA mixed with 10% $CaCl_2$ (25.40 ± 5.58 minutes) at $p < 0.0001$.

Our study is in agreement with the previously mentioned studies that concluded that a significant reduction in setting time could be accomplished by adding $CaCl_2$ (Abdullah et al., 2002; Bortoluzzi et al., 2009; Lee et al., 2011; Prasad et al, 2015). We have found that the addition of 5%, 10%, or 15% $CaCl_2$ reduced the setting time of all tested materials (EMTA, 20% BIE, and 40% BIE) compared to the same materials without $CaCl_2$. Although adding 15% $CaCl_2$ decreased the setting time of all materials compared to adding 10%, this difference was not statistically significant ($p > 0.05$).

7.3. pH Level

The results of this study showed that all tested materials were strongly basic materials. Their pH levels increased with time and decreased with the addition of $CaCl_2$ at all of the time intervals. Without the addition of $CaCl_2$, at 2 hours, the 40% BIE showed the lowest pH level of 9.01 ± 0.16, followed by 20% BIE (9.70 ± 0.20), and Dycal® (9.90 ± 0.439). The highest pH level observed was in EMTA (10.9 ± 0.31). After 10% and 15% $CaCl_2$ were added to 40% BIE, the material showed the lowest pH level (8.20 ± 0.3, and 8.37 ± 0.33, respectively) among the groups ($p < 0.0001$).

At 28 days, all tested materials showed an increase in the pH levels. Dycal® showed the lowest pH level (11.93 ± 0.27), followed by the 40% BIE + 15 % $CaCl_2$ (12.01 ± 0.07), the 40% BIE + 10 % $CaCl_2$ (12.06 ± 0.082), and the 40% BIE + 5 % $CaCl_2$ (12.08 ± 0.05) without significant difference between them ($p > 0.05$). There was no significant difference in the pH levels of any materials between adding 10% or 15% $CaCl_2$ ($p > 0.05$).

The results of Prasad et al.'s study (2015), showed that after 24 hours immersion of MTA and MTA + 10% $CaCl_2$ samples in 60 ml of deionized distilled water, MTA + 10% $CaCl_2$ showed a statistically lower pH level (11.22 ± 0.15) than MTA (12.54 ± 0.27) at $p < 0.0001$. Another study showed a slight increase in the pH level of MTA and MTA + 10% $CaCl_2$ with time. The pH level of MTA increased from 12.8 ± 0.1 at 3 hours to 12.9 ± 0.1 at 24 hours ($p > 0.05$), and from 11.4 ± 0.2 to 11.5 ± 0.2 for MTA + 10% $CaCl_2$ ($p > 0.05$). The difference in pH levels between both materials were statistically significant at $p < 0.01$ at both times intervals (Lee et. al., 2011).

This study is in agreement with the previously mentioned studies of Prasad et al. (2015) and Lee et al. (2011). We found that the pH levels of all tested materials increased with time, and that adding $CaCl_2$ significantly reduced the pH level; however, its effect diminished over time.

The pH findings of our study contradict the findings of Bortoluzzi et al. (2008). In that study, the researcher found that the pH level of WMTA decreased over time from 9.77 ± 0.18 at 1 day to 7.91 ± 0.12 at 28 days. The addition of 10% $CaCl_2$ to WMTA initially elevated the pH level to 10.06 ± 0.13 at 1 day, and the mixture had an almost comparable pH level to WMTA (7.85 ± 0.13) at 28 days. A possible reason for this difference in the results is that in Bortoluzzi's study, the same samples used for testing the pH levels were also used for measuring solubility; hence the materials were subjected to removal and dehydrations between time intervals. This could have resulted in material loss; thus the proportion of material weight to water was not constant throughout the study. Also, the WMTA materials were used instead of GMTA, and the pH meter were just calibrated with a buffer solution of pH 7.0.

The addition of calcium-based electrolytes ($CaCl_2$) tended to be acidic after dissolving and suppressing ionization of $Ca(OH)_2$; thus the percentage of dissociation of $Ca(OH)_2$ decreased and resulted in decreased pH levels because of the common ion effect. However, this decline in pH levels at an alkaline condition, might not have affected the antimicrobial property of the materials (Dian-Yu Ji et al, 2011; Prasad et al., 2015).

7.4. Compressive Strength

The result of our study showed that at 24 hours, Dycal® had the weakest strength of 10.78 ± 1.15 MPa, followed by EMTA (29.62 ± 3.03 MPa), 20% BIE (32.16 ± 4.76 MPa), and 40%BIE had the strongest (32.916 ± 4.76 MPa; $p < 0.0001$). All materials showed an increase in strength over time. However, at 28 days, Dycal® remained the weakest material (19.011 ± 3.325 MPa) and 40% BIE remained the strongest (55.135 ± 8.850 MPa). There was a linear relation with $CaCl_2$ concentration and reducing of the material's strength.

Hui-gang Xiao (2004) and Akbari et al. (2013) found that adding SiO_2 to Portland cement induced resistance to compression, especially at the early stages (3 days). In the study of Akbari et al. the compressive strength of the three materials (MTA, 8% SiO_2 + MTA, and 10% SiO_2 + MTA) were compared using a universal testing machine (Instron, Zwick, Germany), at 1 day and at 7 days. The results showed that the compressive strengths of 8% SiO_2 + MTA (2.7 ± 0.66) and 10% SiO_2 + MTA (1.92 ± 1.29 MPa) were higher than that of EMTA (1.16 ± 0.31 MPa). The strength of all materials increased over time (2.75 ± 0.81 MPa, 2.39 ± 0.52 MPa, and 2.19 ± 0.87 MPa, respectively). However, this difference was not statistically significant ($p > 0.05$).

The same finding has been achieved in this study, where the compressive strength of EMTA (29.62 ± 3.03 MPa) was statistically lower than silica-containing materials 20% BIE (32.16 ± 4.76 MPa) and 40% BIE (36.91 ± 5.04 MPa) at 1 day. The compressive strength of all materials increased over time at day 28 to 44.01 ± 7.49 MPa for the EMTA, 44.88 ± 3.55 MPa for 20% BIE, and 55.13 ± 8.85 MPa for the 40% BIE.

Prasad et al., (2015) studied the effect of adding $CaCl_2$ to MTA at 24 hours, 3 days, and 7 days. The test methodology the researchers used was ISO 9917, using a universal testing machine (Instron 1195, Norwood, MA, USA) with a crosshead speed of 1.0 mm/min. One hundred samples were prepared from a mold that was 4.0 mm in diameter and 6.0 mm in height, and they were immersed in distilled water for the time intervals. Prasad et al. found an increase of compressive strength of all materials over time, including a statistically inverse relationship with $CaCl_2$. The compressive strength of MTA material was 18.40 ± 0.64 MPa at day 1 and 36.24 ± 3.33 MPa at day 7. On the other hand, the strength of MTA + 10% $CaCl_2$ decreased to 10.82 ± 1.08 MPa and 33.37 ± 3.18 MPa at days 1 and 7 respectively. In this study we found the same trend of increasing compressive strength over time and that $CaCl_2$ reduced the strength of the materials; however, the magnitude of strength in our study was higher than Prasad et al.'s study. Our findings showed that MTA's compressive strength was 29.620 ± 3.038 MPa at day1 and increased to 32.143 ± 5.295 at day 7. On the other hand, the compressive strength of MTA + 10% $CaCl_2$ increased from 21.81 ± 3.62 MPa at day 1 to 23.629 ± 4.629 MPa at day 7. One possible explanation of the difference in the magnitude of the compressive strength of the MTA and the MTA + 10% $CaCl_2$ between this study and Prasad et al.'s study, is that in Prasad et al.'s study, samples were stored in distilled water for the tested time intervals. On the other hand, in this study samples were stored at 37°C with 100% relative humidity, and not immersed in water. Storing the specimens in water could result in water penetration; weaken the bond strength within the material and loss of material substance. The effect of $CaCl_2$ on decreasing the compressive strength was also documented in the study of Lee BN in 2011.

Although the addition of $CaCl_2$ decreased the setting time of all tested materials, it inversely affected the compressive strength of the tested materials. This could be resulted from the hygroscopic expansion of the material after adding $CaCl_2$, which might have induced tension to the final mass, and weaken the bonds strength (Machado et. al., 2010).

One of the limitations of this study was that the compressive strength of the materials was not tested after the materials were immersed in deionized water for measuring solubility. Adding $CaCl_2$ at any concentrations increased the material solubility; thus, a concern exists regarding the remaining strength of the material after dissolving the $CaCl_2$.

7.5. Solubility

The solubility results of this study showed that over 28 days, Dycal® was the most soluble (30.768 ± 1.68%) among the tested materials without $CaCl_2$ ($p < 0.0001$), followed by EMTA (1.75 ± 0.61%), and 20% BIE (1.49 ± 0.35%). The 40% BIE was the least soluble with 0.93 ± 0.29% weight loss at $p < 0.0001$. Adding $CaCl_2$ to all materials increased the material solubility and there is no statistical significant difference between adding 10% and 15% $CaCl_2$ ($p > 0.05$).

There exists a debate among investigators in defining the degree of solubility of MTA. The majority of studies reported low or no solubility for MTA; however, an increase in solubility over time has been reported by some researchers (Islam & Yap, 2006; Poggio et al., 2007; Torabinejad et al., 1995(a)).

Different factors such as powder-to-water ratio could influence the degree of solubility of the materials. Increasing the water increased calcium hydroxide release from MTA. The addition of bismuth oxide (which is insoluble in water) to MTA is another factor that plays a role in decreasing the solubility of MTA (Fridland & Rosado, 2003). In addition, adding $CaCl_2$ to MTA significantly increased its solubility (Bortoluzzi et al., 2006). A possible explanation of increasing the solubility after adding $CaCl_2$, is that the hygroscopic property of the compound and thus may have absorbed more water from the environment during cement preparation. The media of testing solubility also influences the results. Lower solubility of MTA material was observed when the material was placed in Synthetic Tissue Fluid (STF) compared to material placed in deionized water. This could be attributed to the

higher concentration of ions in STF compared to deionized water, which results in lower fluid penetration into the bulk of MTA (Saghiri et al., 2011).

Our study supports other previously mentioned studies in showing the minimal solubility of EMTA that also increases over time from 1, 7, 21 to 28 days (0.552 ± 0.351%, 0.912 ± 0.471%, 1.229 ± 0.522%, and 1.495 ± 0.611%, respectively).

Our solubility findings contradict the study of Bortoluzzi et al. (2008). The study found that the solubility of WMTA remained constant at 15% (from 1 to 28 days); however, that study is in agreement with our study's conclusion that the solubility EMTA in our increased when 10% $CaCl_2$ were added. However, the increase in the level of solubility in this study appeared over time not at day1 (9.33% at day 1 and 19.50% at day 28).

High solubility of the material indicates weak chemical bonds, strong ion solvent interaction, and more porous structure. Thus, a material with high solubility is associated with low strength in the present of solvent.

CONCLUSIONS

8. CONCLUSION

Many different materials have been used for pulp capping. Most commonly used are calcium hydroxide (Dycal®), and mineral trioxide aggregate (MTA). However, none of the available pulp capping material has the property of induce dentinogenesis. The present study has proposed a new alternative pulp capping material containing bioactive inorganic element (BIE). BIE is a bioactive glass composed of silicon, calcium and phosphorous. The materials have been developed and studied for biocompatibility in tissue engineering, bone regeneration, and odontogenic properties at Boston University. The aim of this study was to test the physical and mechanical properties (setting time, pH level, compressive strength, and solubility) of two groups of BIE-containing materials: 20% BIE (20% BIE, 60% Portland cement, and 20% bismuth oxide) and 40% BIE (40% BIE, 40% Portland cement, and 20% bismuth oxide); and to compare them to calcium hydroxide (Dycal®) and experimental mineral trioxide aggregate (EMTA) with and with out calcium chloride at three different concentrations (5%, 10%, and 15%).

Within the conditions and limitations of the study, the following results can be concluded:

For the setting time, the results of comparing the mean setting time of the materials without adding $CaCl_2$ (Dycal®, EMTA, 20% BIE, and 40% BIE) showed that Dycal® had statistically the shortest setting time (5.80 ± 1.13 minutes; $p < 0.0001$), followed by 40% BIE

then 20% BIE without significant difference between the setting time of both materials (60.00 ± 6.66, 67 ± 13.37 minutes; $p > 0.05$). c

The addition of 5%, 10% and 15% $CaCl_2$ significantly reduced the setting time of all tested materials tested without $CaCl_2$ ($p < 0.0001$). However, there was no significant difference between adding 10% and 15% $CaCl_2$ ($p > 0.05$). The 40% BIE materials with 10% and 15% $CaCl_2$ had a comparable setting time (14.00 ± 2.10 minutes and 9.40 ± 0.96 minutes, respectively) to Dycal® (5.80 ± 1.13 minutes) with no significant difference in the mean setting time ($p > 0.05$).

The results of comparing the pH level between groups showed that all tested materials had a high alkaline level that increased with time, and decreased with the addition of $CaCl_2$. For materials without $CaCl_2$, at all time intervals the EMTA material showed the highest pH level followed by the 20% BIE, then the 40% BIE, and the Dycal® material showed the lowest pH level. After the addition of $CaCl_2$, the pH level of all materials decreased, with no significant difference between adding 10% and 15% $CaCl_2$ to any tested materials at all time intervals ($p > 0.05$).

At 28 days, the pH level of Dycal® (11.93 ± 0.27) was comparable to the pH level of 40% BIE (12.10 ± 0.04), 40% BIE + 5% $CaCl_2$ (12.08 ± 08), 40% + 10% $CaCl_2$ (12.06 ± 0.28), and 40% BIE + 15% $CaCl_2$ (12.01 ± 0.07) with no significant difference between them $p > 0.05$.

Regarding compressive strength, 40% BIE had the highest compressive strength among the groups (36.91 ± 5.04 MPa at 1 day, and 55.13 ± 8.85 MPa at day 28), followed by 20% BIE (32.16 ± 4.74 MPa at 1 day, and 44.88 ± 3.55 MPa at day 28), and then EMTA (29.62 ± 3.03 MPa at 1 day, and 44.01 ± 7.49 MPa at day 28). Dycal® had the lowest compressive strength during all time intervals (10.78 ± 1.15 MPa at 1 day, and 19.76 ± 3.32 MPa at 28 days). The difference between the compressive strength of Dycal® and the rest of tested materials was significant at $p < 0.0001$. The compressive strength of all tested materials increased over time and reduced with the addition of 10% and 15% $CaCl_2$ ($p < 0.0001$).

In the tests for solubility, the results showed that solubility increased with time and with the addition of $CaCl_2$. Dycal® was the most soluble material ($p <0.0001$) during all time intervals (8.54 ± 0.67 % at 1day, and increased to 30.76 ± 1.67 % at 28 days). The solubility levels of EMTA (0.55 ± 0.31 % at 1day, and increased to 1.75 ± 0.61 % at 28 days), 20% BIE (0.53 ± 0.22 % at 1day, and increased to 1.49 ± 0.35 % at 28 days), and 40 % BIE (0.29 ± 0.21 % at 1day, and increased to 0.93 ± 0.29 % at 28 days) were comparable during all time intervals ($p > 0.05$). Adding 5% of $CaCl_2$ or greater significantly increased the materials solubility at all time intervals ($p < 0.0001$).

Within the limitations of this study, the results showed that material containing 40% BIE have promising results regarding their physical and mechanical properties compared to Dycal® and EMTA. It has a comparative level of setting time and pH level with Dycal® after adding 10% or 15% of $CaCl_2$, and they have superior compressive strength and lower

solubility than Dycal® and EMTA. Therefore, the 40% BIE with 10% $CaCl_2$ could compete the Dycal® and EMTA when used in pulp capping.

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APPENDIX

9. APPENDIX

Table 10-27: Raw data of the depth of penetration of the Dycal® groups vs. time.

Control, Dycal®										
Specimen	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Time (min)		**Depth of Penetration (mm)**								
2	0.032	0.029	0.03	0.027	0.013	0.019	0.024	0.089	0.029	0.04
4	0.017	0.019	0.015	0.013	0.011	0.011	0.01	0.012	0.014	0.009
6	0.01	0.009	0.012	0.009	0.005	0.005	0.003	0.006	0.007	0.008
8	0.003	0.003	0.002	0.002	0.001	0.002	0.002	0.001	0.005	0.006
10	0	0	0	0	0	0	0	0	0	0

Table 10-28: Raw data of the depth of penetration of the EMTA groups vs. time.

EMTA										
Specimen	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Time (min)		**Depth of Penetration (mm)**								
2	2.28	2.24	2.26	3.187	2.829	3.222	3.884	3.884	3.134	2.776
4	1.38	2.16	2.57	3.059	1.585	2.389	3.349	3.349	2.893	2.755
6	1.3	1.47	2.49	2.77	1.791	2.227	3.487	3.487	2.554	2.123
8	0.907	1.44	1.58	2.337	1.478	1.808	2.677	2.677	2.432	2.333
10	0.569	1.4	0.872	1.68	1.158	1.345	2.532	2.532	1.883	2.1
15	0.306	0.681	0.373	0.932	0.712	1.1	1.898	1.898	1.257	1.736
20	0.399	0.619	0.125	0.712	0.651	0.987	0.278	0.278	1.077	1.148
30	0.105	0.403	0.157	0.489	0.217	0.422	0.136	0.136	0.405	0.934
40	0.054	0.207	0.72	0.237	0.167	0.325	0.133	0.133	0.301	0.343
50	0.048	0.107	0.069	0.145	0.123	0.149	0.079	0.079	0.228	0.153
60	0.028	0.089	0.063	0.089	0.086	0.131	0.022	0.022	0.147	0.109
70	0.02	0.076	0.043	0.056	0.048	0.122	0.047	0.047	0.074	0.055
80	0.015	0.042	0.025	0.029	0.028	0.066	0.031	0.031	0.033	0.041
90	0.007	0.019	0.014	0.022	0.016	0.032	0.022	0.022	0.019	0.033
110	0.008	0.015	0.008	0.013	0.015	0.021	0.011	0.011	0.009	0.017
120	0.002	0.007	0.004	0.003	0.008	0.016	0	0	0	0.006
130	0	0.001	0	0	0.002	0.006	0	0	0	0
140	0	0	0	0	0	0	0	0	0	0

Table 10-29: Raw data of the depth of penetration of the EMTA+5% $CaCl_2.2H_2O$ groups vs. time

EMTA+5% $CaCl_2.2H_2O$										
Specimen	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Time (min)	**Depth of Penetration (mm)**									
2	3.777	3.666	3.444	3.652	3.781	3.443	3.224	3.882	3.784	3.515
4	3.278	3.127	3.31	3.205	3.157	3.127	3.068	3.124	3.178	3.154
6	2.678	2.433	2.109	1.98	1.998	2.421	2.033	2.113	2.804	2.556
8	1.728	1.511	1.554	1.555	1.332	1.864	1.731	1.814	1.871	1.813
10	0.998	0.955	1.109	0.943	1.008	1	0.731	0.998	0.957	0.983
15	0.663	0.83	0.988	0.651	0.661	0.551	0.25	0.253	0.329	0.388
20	0.445	0.211	0.561	0.117	0.338	0.331	0.355	0.25	0.266	0.281
30	0.169	0.016	0.114	0.066	0.045	0.228	0.05	0.088	0.118	0.045
40	0.027	0.035	0.081	0.021	0.022	0.019	0.028	0.03	0.033	0.018
50	0.014	0.009	0.021	0.011	0.015	0.001	0.02	0.01	0.011	0.013
60	0.008	0	0.006	0.003	0.002	0	0.005	0.002	0.003	0.008
70	0.001	0	0	0	0	0	0	0	0	0
80	0	0	0	0	0	0	0	0	0	0

Table 10-30: Raw data of the depth of penetration of the EMTA+10% $CaCl_2.2H_2O$ groups vs. time

EMTA+10% $CaCl_2.2H_2O$										
Specimen	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Time (min)	**Depth of Penetration (mm)**									
2	3.784	3.783	3.718	3.816	3.771	3.722	3.776	3.766	3.878	3.666
4	3.023	2.718	2.701	3.753	3.569	3.227	2.897	3.561	3.035	3.158
6	0.734	0.541	0.367	2.254	2.02	1.92	1.876	1.48	1.121	1.11
8	0.217	0.145	0.158	0.333	0.308	0.3	0.186	0.063	0.101	0.053
10	0.06	0.08	0.057	0.13	0.06	0.064	0.05	0.13	0.025	0.015
15	0.013	0.012	0.013	0.03	0.02	0.015	0.012	0.014	0.01	0.009
20	0.004	0	0.003	0.007	0.017	0.007	0.004	0.001	0	0
30	0	0	0	0	0.008	0	0	0	0	0
40	0	0	0	0	0	0	0	0	0	0

Table 10-31: Raw data of the depth of penetration of the EMTA+15% $CaCl_2.2H_2O$ groups vs. time

EMTA+15% $CaCl_2.2H_2O$										
Specimen	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Time (min)	**Depth of Penetration (mm)**									
2	3.9	4	4.028	3.77	3.649	3.811	3.876	3.572	3.781	3.765
4	3.778	3.89	3.963	3.741	3.456	3.8	3.687	3.412	3.76	3.588
6	1.67	2.817	3.001	3.646	3.385	3.303	3.167	0.864	0.598	0.881
8	1.2	0.54	0.4	1.687	1	0.66	0.987	0.106	0.101	0.177
10	0.221	0.187	0.107	1.798	0.819	1.016	0.194	0.035	0.025	0.05
15	0.02	0.021	0.017	0.025	0.018	0.019	0.028	0.008	0.01	0.003
20	0.002	0.007	0.005	0	0.001	0	0.005	0.001	0.009	0
30	0	0	0	0	0	0	0	0	0	0

Table 10-32: Raw data of the depth of penetration of the 20% BIE groups vs. time.

20% BIE										
Specimen	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Time (min)		**Depth of Penetration (mm)**								
2	3.332	3.004	4.032	3.115	2.884	2.789	3.054	3.547	2.997	3.008
4	3.118	3.121	3.879	2.341	2.301	2.283	3	2.773	1.765	2.784
6	2.527	2.609	2.529	1.475	1.839	1.228	2.727	2.378	1.094	1.855
8	1.356	1.552	1.733	0.94	1.388	0.854	1.811	1.644	0.705	0.893
10	1.132	0.847	0.671	0.453	1.155	0.468	0.438	0.905	0.384	0.551
15	0.321	0.396	0.463	0.166	0.483	0.164	0.337	0.134	0.142	0.216
20	0.165	0.183	0.14	0.108	0.148	0.078	0.085	0.072	0.044	0.166
30	0.063	0.05	0.07	0.049	0.057	0.056	0.072	0.048	0.032	0.055
40	0.059	0.063	0.041	0.015	0.033	0.04	0.024	0.027	0.026	0.042
50	0.026	0.048	0.028	0.006	0.028	0.037	0.012	0.015	0.001	0.033
60	0.016	0.035	0.002	0	0.020	0.022	0.002	0.007	0	0.02
70	0.005	0.029	0	0	0.003	0.018	0	0	0	0.012
80	0	0.022	0	0	0	0.008	0	0	0	0.0008
90	0	0.01	0	0	0	0	0	0	0	0
110	0	0	0	0	0	0	0	0	0	0

Table 10-33: Raw data of the depth of penetration of the 20% BIE+5% $CaCl_2.2H_2O$ groups vs. time

20% BIE+5% CaCl2.2H2O										
Specimen	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Time (min)	**Depth of Penetration (mm)**									
2	2.933	3.004	2.764	2.351	2.895	2.553	2.079	2.775	2.367	2.221
4	2.64	2.754	2.043	1.925	2.224	2.464	1.997	2.478	2.114	2.128
6	1.356	1.744	1.027	0.627	1.065	1.267	1.006	1.005	1.778	1.007
8	1.185	1.027	1.006	0.195	0.168	0.263	0.281	0.484	0.656	0.278
10	0.534	0.87	0.088	0.022	0.023	0.094	0.178	0.298	0.448	0.053
15	0.053	0.348	0.023	0.016	0.02	0.025	0.042	0.049	0.199	0.021
20	0.016	0.077	0.013	0.01	0.018	0.016	0.013	0.022	0.049	0.011
30	0.007	0.005	0.007	0.009	0.01	0.002	0.005	0.012	0.013	0.002
40	0	0	0	0	0.001	0	0	0.001	0.001	0
50	0	0	0	0	0	0	0	0	0	0

Table 10-34: Raw data of the depth of penetration of the 20% BIE+10% $CaCl_2.2H_2O$ groups vs. time.

20% BIE+10% $CaCl_2.2H_2O$										
Specimen	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Time (min)	**Depth of Penetration (mm)**									
2	3.18	3.454	3.554	3.332	3.005	3.22	3.58	3.448	3.551	3.665
4	2.808	3.088	3.088	2.878	2.88	2.816	2.998	2.088	2.078	3.122
6	1.889	2.553	2.551	0.988	1.789	1.889	2.008	1.887	1.885	2.075
8	0.889	1.005	2.004	0.335	0.689	0.789	1.056	1.009	1.002	1.778
10	0.557	0.674	1.545	0.112	0.211	0.557	0.877	0.774	0.574	1.041
15	0.051	0.033	0.588	0.01	0.032	0.042	0.091	0.041	0.023	0.071
20	0.001	0.001	0.114	0	0.001	0.001	0.051	0.001	0.01	0.033
30	0	0	0.01	0	0	0	0.001	0	0	0.001
40	0	0	0	0	0	0	0	0	0	0

Table 10-35: Raw data of the depth of penetration of the 20% BIE+15% $CaCl_2.2H_2O$ groups vs. time.

20% BIE+15%										
Specimen	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Time (min)	**Depth of Penetration (mm)**									
2	3.11	2.889	2.878	2.988	3.211	2.778	3.033	2.278	3.122	2.775
4	2.8	1.753	2.004	1.835	2.732	1.112	2.558	1.888	2.075	1.978
6	1.589	1.001	1.645	1.012	0.995	0.445	2.033	1.002	0.841	1.044
8	0.689	0.674	0.585	0.088	0.553	0.167	1.722	0.874	0.071	0.081
10	0.237	0.0225	0.114	0.021	0.078	0.021	1.117	0.027	0.023	0.033
15	0.01	0.001	0.01	0.01	0.026	0.008	0.015	0.01	0.001	0.014
20	0	0	0	0	0.01	0	0.008	0	0	0.001
30	0	0	0	0	0	0	0	0	0	0

Table 10-36: Raw data of the depth of penetration of the 40% BIE groups vs. time

40% BIE										
Specimen	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Time (min)		**Depth of Penetration (mm)**								
2	2.578	2.881	2.899	4.469	4.005	3.843	2.986	2.663	2.922	2.877
4	2.565	2.273	2.322	4.329	3.745	3.577	2.314	1.953	2.114	2.122
6	2.421	1.825	2.135	3.241	3.543	3.099	1.006	1.231	1.723	1.511
8	1.442	0.629	1.884	2.726	3.048	3.034	0.964	0.737	0.976	0.922
10	0.748	0.208	0.428	0.877	1.929	2.823	0.268	0.309	0.72	0.603
15	0.214	0.082	0.174	0.406	0.89	1.003	0.169	0.142	0.603	0.351
20	0.079	0.045	0.062	0.221	0.133	0.796	0.063	0.067	0.261	0.246
30	0.04	0.035	0.042	0.127	0.077	0.331	0.057	0.063	0.147	0.061
40	0.035	0.02	0.037	0.02	0.049	0.105	0.048	0.035	0.057	0.052
50	0.021	0.016	0.021	0.011	0.011	0.046	0.009	0.008	0.035	0.028
60	0.017	0.005	0.006	0.003	0.001	0.029	0	0	0.011	0.025
70	0.007	0	0	0	0	0.008	0	0	0	0
80	0	0	0	0	0	0	0	0	0	0

Table 10-37: Raw data of the depth of penetration of the 40% BIE+5% $CaCl_2.2H_2O$ groups vs. time

40% BIE+5% $CaCl_2.2H_2O$										
Specimen	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Time (min)	**Depth of Penetration (mm)**									
2	2.828	2.667	2.779	2.667	2.775	2.767	2.665	2.775	2.885	2.776
4	1.737	1.889	2.005	1.866	2.008	1.779	1.332	1.901	1.996	1.565
6	0.221	1.004	1.774	1.012	1.411	0.887	0.668	1.357	0.664	0.884
8	0.088	0.779	1.337	0.774	0.885	0.221	0.367	0.973	0.227	0.211
10	0.021	0.221	1.036	0.132	0.041	0.112	0.029	0.28	0.014	0.188
15	0.018	0.01	0.025	0.188	0.017	0.013	0.016	0.032	0.015	0.007
20	0	0	0.003	0.007	0.003	0	0.008	0.01	0.008	0
30	0	0	0	0	0	0	0	0	0	0

Table 10-38: Raw data of the depth of penetration of the 40% BIE+10% $CaCl_2.2H_2O$ groups vs. time

40% BIE+10% $CaCl_2.2H_2O$										
Specimen	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Time (min)	**Depth of Penetration (mm)**									
2	2.808	2.331	2.441	2.678	2.688	2.816	2.408	2.448	2.551	2.337
4	1.089	1.033	1.088	0.988	1.789	1.889	1.056	1.088	1.885	1.041
6	0.889	0.665	0.588	0.335	0.689	0.557	0.877	0.774	1.002	0.071
8	0.087	0.033	0.114	0.112	0.211	0.042	0.091	0.221	0.574	0.033
10	0.051	0.001	0.081	0.033	0.032	0.001	0.051	0.041	0.023	0.017
15	0.001	0	0.01	0.01	0.001	0	0.001	0.001	0.01	0.004
20	0	0	0	0	0	0	0	0	0	0

Table 6-39: Raw data of the depth of penetration of the 40% BIE+15% $CaCl_2.2H_2O$ groups vs. time

40% BIE+15% $CaCl_2.2H_2O$										
Specimen	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Time (min)	**Depth of Penetration (mm)**									
2	2.077	2.011	2.221	2.003	2.221	1.988	2.553	2.441	2.331	2.441
4	0.114	0.668	1.044	0.558	0.883	0.669	0.558	0.887	0.779	0.773
6	0.066	0.156	0.331	0.05	0.054	0.332	0.018	0.118	0.179	0.086
8	0.01	0.051	0.044	0.01	0.018	0.061	0.006	0.021	0.055	0.033
10	0	0.008	0.008	0.007	0.004	0.01	0	0.004	0.017	0.004
15	0	0	0	0	0	0	0	0	0.007	0
20	0	0	0	0	0	0	0	0	0	0

Table 10-40: pH at 2 hours of all tested materials

Specimen / Material	1	2	3	4	5	6	7	8	9	10
Dycal®	10.3	10.1	10	9.9	10.7	9.7	9.7	9.1	9.5	10
EMTA	10.7	11.16	11.22	10.25	11.2	10.94	11.18	10.8	11.01	10.6
20%BIE	9.5	9.3	9.8	9.7	9.5	9.8	9.9	9.8	9.9	9.8
40% BIE	9.3	8.9	9	8.9	9	9	8.9	8.8	9	9.3
EMTA + 5% $CaCl_2$	10.4	10.2	10.5	10.7	10.4	10.2	10.8	10.5	10.3	10.7
EMTA + 10% $CaCl_2$	9.4	9.7	10.25	10.66	9.82	10.33	10.61	10.53	9.89	10.43
EMTA+ 15% $CaCl_2$	9.4	9.7	10.25	10.66	9.82	10.33	10.3	10.53	9.89	10.43
20%BIE + 5% $CaCl_2$	9.5	9.3	9.5	9.7	9.5	9.8	9.7	9.4	9.5	9.5
20%BIE+ 10% $CaCl_2$	9.2	8.8	9.3	8.9	9	9	8.9	8.8	9.4	9.3
20%BIE+15% $CaCl_2$	9.2	8.8	9.3	8.9	9	9	8.9	8.8	8.8	9
40%BIE + 5% $CaCl_2$	9.1	8.8	9	8.9	8.9	9	8.9	8.88	8.8	9
40%BIE+ 10% $CaCl_2$	8.6	7.88	8.54	8.7	8.6	7.88	8.5	7.9	8.5	8.6
40%BIE+ 15% $CaCl_2$	8.55	7.88	8.54	7.88	8.31	7.9	8.5	7.9	8.5	8.11

Table 10- 41: pH at 24 hours of all tested materials

Specimen / Material	1	2	3	4	5	6	7	8	9	10
Dycal®	10.7	10.5	10.7	10.4	11.1	10.5	10.4	10.6	10.8	11.1
EMTA	11.85	11.95	12.09	11.96	12.03	12.04	12.08	11.79	11.75	11.91
20%BIE	11.2	11.1	11.1	11.2	11.1	11.1	11.2	10.9	10.9	11.2
40% BIE	12	12	12	12.1	12	11.9	11.6	11.5	11.8	11.8
EMTA + 5% $CaCl_2$	10.8	10.9	11	11.2	11.2	11.1	10.8	10.5	10.3	10.7
EMTA + 10% $CaCl_2$	10.29	10.11	10.83	9.86	11.19	10.77	11.3	10.4	10.79	11.01
EMTA+ 15% $CaCl_2$	9.4	10.55	10.74	10.66	10.99	10.33	10.61	10.53	10.79	10.43
20%BIE + 5% $CaCl_2$	11.2	11.1	11.1	11.2	11.1	11.1	11.2	10.8	10.9	11.2
20%BIE+ 10% $CaCl_2$	10.5	10.7	9.9	11.1	11	11	10.8	10.7	10.8	10.5
20%BIE+15 % $CaCl_2$	10.5	10.7	9.9	10.7	10.5	10.88	10.8	10.7	10.8	10.5
40%BIE + 5% $CaCl_2$	11.69	11.5	11.46	11.58	11.67	11.46	11.59	11.58	11.55	11.58
40%BIE+ 10% $CaCl_2$	10.5	10.7	10.4	10.7	10.5	10.88	10.55	10.7	10.8	10.5
40%BIE+ 15% $CaCl_2$	10.5	10.7	10.4	10.7	10.5	10.44	10.8	10.7	10.5	10.5

Table 10-42: pH at 7 days of all tested materials

Specimen / Material	1	2	3	4	5	6	7	8	9	10
Dycal®	12	11.4	11.8	11.6	12	11.5	12	11.7	11.9	11.9
EMTA	12.37	12.46	12.37	12.38	12.42	12.38	12.48	12.38	12.38	12.37
20%BIE	11.56	11.47	11.44	11.42	11.42	11.43	11.4	11.45	11.57	11.6
40% BIE	12	11.98	11.99	12.05	11.99	12	11.99	11.97	11.98	11.98
EMTA + 5% $CaCl_2$	11.96	11.8	11.86	12.08	11.91	11.6	12.12	11.63	12.1	12.17
EMTA + 10% $CaCl_2$	11.3	11.5	11.5	11.3	11.5	11.6	11.2	11.6	11	11.4
EMTA+ 15% $CaCl_2$	11.2	10.9	11.3	11.3	11.2	11.2	11.1	10.9	11.1	11
20%BIE + 5% $CaCl_2$	11.25	10.98	11.39	11.48	11.49	11.2	11.67	11.31	11.38	11.62
20%BIE+ 10% $CaCl_2$	11	11.21	11.22	11	11.11	11	11.2	11	11.22	11.28
20%BIE+15 % $CaCl_2$	11	11.21	10.88	11	11.11	11	10.9	11	11.22	10.88
40%BIE + 5% $CaCl_2$	12.02	11.91	11.85	11.6	11.84	11.8	11.94	11.89	11.86	11.96
40%BIE+ 10% $CaCl_2$	11.09	10.9	11.07	11.13	10.85	11.64	11.07	11.14	10.93	11.05
40%BIE+ 15% $CaCl_2$	10.7	10.7	10.9	11.4	11.33	10.91	11.3	11.3	10.88	10.9

Table 10-43: pH at 21 days of all tested materials

Specimen / Material	1	2	3	4	5	6	7	8	9	10
Dycal®	12.2	11.7	11.8	11.7	12.1	11.7	11.4	11.9	12	12
EMTA	12.53	12.31	12.46	12.43	12.41	12.54	12.45	12.5	12.44	12.46
20%BIE	11.8	11.7	11.7	11.7	11.9	11.8	11.8	11.9	11.9	12
40% BIE	12.06	12.02	12.07	12.14	12.18	12.06	12.11	12.02	12.11	12.09
EMTA + 5% $CaCl_2$	12.38	12.38	12.5	12.38	12.37	12.37	12.33	12.29	12.47	12.32
EMTA + 10% $CaCl_2$	12	12.1	11.9	12.1	12.1	12.2	11.9	11.8	12.2	12.1
EMTA+ 15% $CaCl_2$	11.82	12	12.2	12.1	12	12	11.64	12.2	12.1	12.2
20%BIE + 5% $CaCl_2$	12.41	12.36	12.34	12.41	12.36	12.36	12.31	12.25	12.26	12.37
20%BIE+ 10% $CaCl_2$	11.67	11.65	11.92	11.7	11.66	11.77	11.82	11.77	11.66	11.79
20%BIE+15 % $CaCl_2$	11.44	11.47	11.44	11.42	11.42	11.43	11.4	11.45	11.42	11.47
40%BIE + 5% $CaCl_2$	12.09	11.99	12.07	11.91	12.17	11.89	12.11	12.17	12.03	12.28
40%BIE+ 10% $CaCl_2$	11.56	11.47	11.44	11.42	11.42	11.43	11.4	11.45	11.57	11.6
40%BIE+ 15% $CaCl_2$	11.11	11	11.2	11.6	11.6	11.5	11.3	11.3	11.4	11.2

Table 10-44: pH at 28 days of all tested materials

Specimen / Material	1	2	3	4	5	6	7	8	9	10
Dycal®	12.1	11.7	11.6	11.8	12	12.5	11.6	11.9	12.1	12
EMTA	12.53	12.49	12.46	12.5	12.43	12.49	12.47	12.43	12.59	12.47
20%BIE	12.42	12.4	12.43	12.53	12.43	12.5	12.53	12.51	12.43	12.57
40% BIE	12.09	12.12	12.2	12.06	12.11	12.1	12.11	12.15	12.02	12.08
EMTA + 5% $CaCl_2$	12.36	12.37	12.33	12.41	12.32	12.38	12.38	12.44	12.31	12.5
EMTA + 10% $CaCl_2$	12.1	12	12.2	12.1	12	12	12.2	12.2	12.1	12.2
EMTA+ 15% $CaCl_2$	12.1	12	12.2	12.1	12.2	12	12	12.2	12.1	12
20%BIE + 5% $CaCl_2$	12.41	12.3	12.35	12.36	12.27	12.31	12.32	12.32	12.46	12.4
20%BIE+ 10% $CaCl_2$	12.17	12.15	12.11	12.15	12.13	12.08	12.09	12.08	12.11	12.1
20%BIE+15 % $CaCl_2$	12.11	12.15	12.11	12.09	12.13	12.08	12.09	12.08	12.11	12.1
40%BIE + 5% $CaCl_2$	12.06	12.02	12.07	12.14	12.18	12.06	12.11	12.02	12.09	12.09
40%BIE+ 10% $CaCl_2$	12.22	12.22	12.2	12.14	12.13	12.11	12.04	12.11	12.16	11.28
40%BIE+ 15% $CaCl_2$	12	11.98	11.99	12.22	11.99	12	11.99	11.97	12	11.98

Table 10-45: Compressive strength of Dycal vs. time.

Dycal® Sample	1 Day (MPa)	7 Days (MPa)	21Days (MPa)	28 Days (MPa)
1	11.381	10.362	13.635	16.280
2	11.383	11.910	12.616	15.142
3	11.416	14.660	11.984	19.476
4	12.998	13.258	15.288	19.110
5	9.323	12.177	14.075	24.055
6	9.869	10.928	16.583	22.248
7	9.244	15.903	18.306	22.681
8	10.694	11.881	17.359	24.117
9	10.233	9.336	17.753	16.524
10	11.315	10.269	17.867	18.041

Table 10-46: Compressive strength of EMTA vs. time.

EMTA Sample	1 Day (MPa)	7 Days (MPa)	21Days (MPa)	28 Days (MPa)
1	28.340	25.535	41.585	44.672
2	30.807	34.767	47.479	52.316
3	32.528	35.169	45.518	44.718
4	33.709	34.017	37.716	57.566
5	31.505	21.807	35.177	41.636
6	31.003	35.314	45.710	40.085
7	30.507	35.599	40.967	35.120
8	23.655	27.478	47.707	35.129
9	26.866	33.566	43.447	38.405
10	27.283	38.184	39.925	50.465

Table 10-47: Compressive strength of EMTA +5% $CaCl_2$ vs. time.

EMTA +5% $CaCl_2$ Sample	1 Day (MPa)	7 Days (MPa)	21Days (MPa)	28 Days (MPa)
1	26.877	22.657	37.696	47.726
2	21.272	29.461	38.000	53.164
3	32.229	35.236	42.006	36.701
4	24.669	29.259	28.464	31.379
5	20.116	21.280	37.294	31.279
6	28.124	34.141	41.537	47.051
7	28.455	34.164	36.407	37.609
8	30.920	35.263	30.780	45.103
9	29.296	33.213	33.907	44.969
10	35.430	31.219	39.529	47.698

Table 10-48: Compressive strength of EMTA +10% $CaCl_2$ vs. time.

EMTA +10% $CaCl_2$ Sample	1 Day (MPa)	7 Days (MPa)	21Days (MPa)	28 Days (MPa)
1	19.842	17.732	30.218	27.848
2	24.586	25.427	30.987	28.473
3	18.089	27.166	26.544	26.795
4	22.822	18.519	24.585	31.061
5	24.307	28.560	24.921	34.667
6	16.047	19.712	28.811	21.538
7	22.607	24.834	24.780	27.411
8	28.466	28.192	21.263	30.059
9	22.276	28.258	29.131	37.042
10	19.111	17.891	36.765	23.270

Table 10-49: Compressive strength of EMTA +15% $CaCl_2$ vs. time.

EMTA +15% $CaCl_2$ Sample	1 Day (MPa)	7 Days (MPa)	21Days (MPa)	28 Days (MPa)
1	15.946	15.628	24.969	29.550
2	13.576	21.096	34.191	28.301
3	17.138	15.917	30.451	36.210
4	14.980	24.527	28.532	34.752
5	15.460	25.571	24.349	27.546
6	15.629	24.401	29.285	25.556
7	15.066	16.913	19.914	21.186
8	11.263	22.681	20.020	27.809
9	11.616	21.810	24.297	22.944
10	12.938	20.117	32.797	31.734

Table 10-50: Compressive strength of 20% BIE vs. time.

20 % BIE Sample	1 Day (MPa)	7 Days (MPa)	21Days (MPa)	28 Days (MPa)
1	30.995	34.264	38.788	43.337
2	37.880	26.867	49.512	44.208
3	33.387	36.906	40.337	45.686
4	30.831	27.784	43.685	44.933
5	32.357	28.729	45.817	40.047
6	27.844	34.854	46.387	39.486
7	24.736	34.931	45.506	46.146
8	40.024	33.376	44.850	44.988
9	29.435	35.774	41.631	48.631
10	34.177	37.828	42.623	51.387

Table 10-51: Compressive strength of 20% BIE+5% $CaCl_2$ vs. time.

20% BIE +5% $CaCl_2$ Sample	1 Day (MPa)	7 Days (MPa)	21Days (MPa)	28 Days (MPa)
1	31.815	27.919	44.603	37.984
2	35.124	22.950	50.505	39.225
3	24.597	32.647	40.370	52.831
4	35.531	23.011	35.239	50.937
5	35.032	34.995	35.048	55.237
6	30.031	33.026	42.728	40.424
7	29.327	34.788	46.598	52.961
8	29.938	36.232	38.595	33.712
9	25.269	27.946	45.853	38.519
10	23.807	37.005	34.565	36.048

Table 10-52: Compressive strength of 20% BIE+10% $CaCl_2$ vs. time.

20% BIE +10% $CaCl_2$ Sample	1 Day (MPa)	7 Days (MPa)	21Days (MPa)	28 Days (MPa)
1	24.812	22.076	29.348	33.020
2	24.959	23.668	32.706	30.950
3	25.422	26.764	35.284	30.460
4	20.346	21.620	35.625	37.485
5	19.765	29.896	35.717	39.982
6	21.117	20.456	29.962	32.836
7	22.501	24.581	28.621	28.847
8	16.549	21.753	28.472	38.585
9	25.914	28.237	29.686	24.579
10	28.733	25.497	29.959	24.217

Table 10-53: Compressive strength of 20% BIE+15% $CaCl_2$ vs. time.

20% BIE +15% $CaCl_2$ Sample	1 Day (MPa)	7 Days (MPa)	21Days (MPa)	28 Days (MPa)
1	16.250	21.965	29.790	32.655
2	20.169	28.038	31.186	40.733
3	21.242	19.342	30.269	35.910
4	22.130	27.404	26.976	25.221
5	20.749	23.498	24.619	29.931
6	15.019	27.082	26.503	35.593
7	14.092	24.501	32.593	24.162
8	18.930	24.829	22.028	26.890
9	16.215	24.230	21.783	23.559
10	20.571	20.205	31.640	34.712

Table 10-54: Compressive strength of 40% BIE vs. time.

4 0% BIE Sample	1 Day (MPa)	7 Days (MPa)	21Days (MPa)	28 Days (MPa)
1	30.527	35.174	43.120	49.638
2	40.690	41.926	49.471	59.506
3	43.244	31.967	42.486	60.072
4	43.919	34.438	43.062	67.465
5	29.620	42.520	48.697	56.315
6	33.417	31.769	40.631	45.014
7	36.379	27.987	43.515	54.768
8	39.831	41.845	40.119	41.870
9	37.838	45.401	45.911	48.871
10	33.735	41.646	43.974	67.837

Table 10-55: Compressive strength of 40% BIE+5% $CaCl_2$ vs. time.

40% BIE +5% $CaCl_2$ Sample	1 Day (MPa)	7 Days (MPa)	21Days (MPa)	28 Days (MPa)
1	27.009	29.435	37.394	36.492
2	27.780	34.842	47.015	45.174
3	27.473	38.665	45.403	30.629
4	27.121	40.322	39.905	45.571
5	33.440	38.798	37.766	47.655
6	36.644	30.841	38.253	47.694
7	37.481	30.545	49.428	47.350
8	29.774	41.781	37.270	49.704
9	39.509	36.004	43.443	47.717
10	33.972	40.923	46.951	45.193

Table 10-56: Compressive strength of 40% BIE+10% $CaCl_2$ vs. time.

40% BIE +10% $CaCl_2$ Sample	1 Day (MPa)	7 Days (MPa)	21Days (MPa)	28 Days (MPa)
1	22.176	19.176	37.412	32.719
2	19.533	27.995	34.748	36.727
3	24.110	26.315	34.000	35.635
4	28.562	25.676	40.717	37.261
5	21.498	21.383	40.950	38.652
6	25.700	25.585	34.775	41.126
7	23.716	23.069	36.728	46.182
8	20.165	25.279	29.357	32.860
9	23.086	27.277	31.356	33.355
10	24.400	33.331	32.703	38.633

Table 10-57: Compressive strength of 40% BIE+15% $CaCl_2$ vs. time.

40% BIE +15% $CaCl_2$ Sample	1 Day (MPa)	7 Days (MPa)	21Days (MPa)	28 Days (MPa)
1	20.399	25.935	23.665	38.586
2	21.803	21.249	31.021	41.039
3	17.628	22.730	27.432	26.586
4	19.383	25.673	21.997	32.728
5	23.150	28.310	35.466	31.945
6	26.758	20.680	32.713	31.672
7	19.242	30.755	25.724	33.661
8	23.762	31.218	32.706	27.643
9	22.997	21.341	29.305	38.447
10	22.937	23.950	32.440	33.471

Vita

10. VITA

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MAHA SAMEER LINJAWI

Citizenship: Saudi
City of birth: Jeddah, Saudi Arabia
Date of Birth: 14th of January 1983
Gender: Female
Marital Status: Married

EDUCATION

- [2011-Expected September 2016] Boston University, Henry M. Goldman School of Dental Medicine.
  Boston, USA
  Certificate of Advanced Graduate Study (CAGS) in Operative Dentistry
  Doctor of Science in Dentistry, Biomaterials and Restorative Dentistry

- [2009 – 2011] Boston University, Henry M. Goldman School of Dental Medicine.
  Boston, USA
  Master of Science in Dentistry (MSD), Dental Public Health candidate

- [2001 - 2007] King Abdul-Aziz University–School of Dentistry.
  Jeddah, SA
  Bachelor in Dental Medicine & Surgery

- [2000 - 2001] King Abdul-Aziz University–School of Science.
  Jeddah, SA
  Honors: (A) Student Honor Certificate

- [1988 - 2000] Dar AL-Bayan School.
  Jeddah, SA
  Elementary, Intermediate, High School Certificate

WORK EXPERIENCE

INTERNSHIP:

Trained and practiced in the following departments:

- [Sep - Dec 2007] King Abdul-Aziz University HospitalJeddah, SA

Comprehensive Care Clinics for Adults and Pediatrics. (3 Months)
Dental Surgery Clinic and Emergency treatment. (I Month)

- [Jan – Feb 2008] King Fahad Armed Forces Hospital Jeddah, SA

Pedodontics, Implant, Prosthodontics, Endodontics Clinics

- [March -April 2008] King Fahd Hospital (MOH). Jeddah, SA

Periodontics, Operative Clinics.
Dental Surgery Clinic.

- [May - July 2008] King Abdul-Aziz University Hospital. Jeddah, SA

Comprehensive Care Clinic for Adults.

RESIDENCY:

[2012 – 2014] Boston University, Henry M. Goldman School of Dental Medicine. Boston, USA
Certificate of Advanced Graduate Study in Operative Dentistry

[2008 – 2009] King Fahad Hospital (MOH). Jeddah, SA
Dental Resident in Periodontics Clinic

CERTIFICATIONS AND LICENSES/EXAMS

- BCLS Certified (July 2014)
- Saudi Council Dental Selection Exam (March 2007)

EXTRACURRICULAR ACTIVITIES

- Part time course at Boston University, Center for English Language and Orientation Programs: Completion of a 12-week course of Academic and Professional Writing April 16, 2010.
- Participated in a number of different community activities (preventive, educational and screening) organized by BU.
- Organizer of the Female Committee Certificate Award in the 19th

Dental technology & Research Conference (2008)

- Member of the Female Registration Committee Certificate Award in the 19th Dental technology & Research Conference (2008)
- Delivered a number of dental presentations at various schools colleges, and hospitals, in Jeddah, Saudi Arabia (2007).

RESEARCH & SCIENTIFIC ACTIVITY

- Co-Author of a Paper Entitled "Factors Affecting Shade Selection"
- Prevalence of Plaque & Gingivitis Among Female Teenagers in Jeddah, SA (Curriculum Cross-Section Study Survey – 2007)
- The Effect of Die Spacer on Extra Coronal Restorations (Articles review - Curriculum)
-

LANGUAGES

- Arabic – native language
- English (Spoken & Written)

SPECIAL SKILLS

- Good Computer Skills using:

1. Microsoft Words, Excel, PowerPoint.
2. SAS and Epi-Info Statistics Package for Data Analysis.

- Active in Community & Public Health Activities