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# Modification of Portland Cement Properties using Glycerin

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## ABSTRACT

**Objective:** The aim of this study was to evaluate the influence of 15% glycerin on the physico-mechanical properties of Portland cement, in an attempt to apply it to mineral trioxide aggregate (MTA).
**Materials and Methods:** Portland cement, mixed with 10 wt% calcium chloride ($CaCl_2$) as an accelerator, was used in this study. The resultant powder was either mixed with distilled water (PC) or 15% glycerin liquid (PCG). The compressive strength, setting time, cohesion time and anti-washout time of the two cements PC and PCG were tested and compared to those obtained by Biodentine (BD). Furthermore, the internal microstructure of the three tested cements (BD, PC and PCG) was evaluated by scanning electron microscope (SEM).
**Results:** BD provided the highest compressive strength followed by PCG then PC. The highest setting and cohesion times were exhibited by PC while there was no significant difference between BD and PCG. Regarding the anti-washout time test, PC exhibited the highest value followed by BD while the lowest value was provided by PCG.
**Conclusions:** Addition of 15% glycerin to Portland cement has improved the setting time, the cohesiveness and the anti-washout ability of the cement with subsequent improvement in the handling properties as well as increasing its compressive strength. This trial is considered to be a successful step which should be performed on Portland cement twin; the MTA.

***Keywords:*** Portland Cement, Glycerin, Compressive Strength, Setting Time, Anti wash-out ability, SEM

## INTRODUCTION

Many years ago, the introduction of mineral trioxide aggregate (MTA), a derivative of Portland cement, in the dental therapy has received a great attention. This is mainly due to its unique biological activity, as it has antibacterial and anti-inflammatory properties. This is primarily due to the increase in the pH value (12.5) of the surrounding environment resulting from the reaction of tricalcium silicate, the main constituent of both MTA and Portland cement, with water to produce calcium hydroxide. Thus, the resultant calcium hydroxide is responsible for the alkaline medium of the cement. Besides, the cement's ability to set in presence of water or blood (i.e. requires moisture to set) makes its use in contact with a moist tissue, such as the pulp, relatively easy. [1,2]

MTA has proven to be a material with several potential clinical applications. This is mainly due to its biocompatibility and its ability to induce mineralization which promotes dental pulp and peri-radicular tissue regeneration. Furthermore, it has a superior sealing property owing to its setting expansion. [2-4] These attractive properties have encouraged the use of MTA as a potential medicament for pulpotomy procedures, capping of pulps with reversible pulpitis, apexification, repair of root perforation and coronal barrier. [5,6] This is in addition to its successful use in the repair of external root resorption and obturation of retained primary teeth. [7]

On the other hand, the main disadvantages of MTA are its extended setting time and difficult handling. This is mainly due to its granular consistency, initial looseness, tendency to washout in presence of excess moisture and, once the mixture starts to dry, it loses its cohesiveness and becomes difficult to handle. This is in addition to the limitation in achieving high strengths, which lies in the inherent porosity of the material. [1,8,9] Since MTA is very similar in composition to Portland cement, they both appear to have the same advantages and disadvantages. [9]

A series of innovative calcium silicates, based on Portland cement, have been recently developed to get use of the alkaline medium provided during its setting. Biodentine™ is a new calcium silicate based cement, similar to MTA, where tricalcium silicate is found as the main cementitious component. [10-12] Therefore, Biodentine exhibits physical and chemical properties similar to those described for Portland cement derivatives. [11] However, according to the manufacturer, it is characterized by obviously shorter setting time and much more user-friendliness. Biodentine has been developed and produced aiming to bring together the high biocompatibility and bioactivity of calcium silicates with enhanced physical properties, thus offering a homogeneous and dense product with maximized strength. [10,12-14]

On the other hand, the highly successful results and the wide spread of the commercially available mineral trioxide aggregate (MTA) during the last few years necessitate carrying out trials to improve its main drawbacks. [8,15] The close similarity in composition and the low price of Portland cement encourage trials which aim at modifying both physical and mechanical properties of Portland cement as a step to be followed by their application on MTA.

In Portland cement industry, attempts were performed to incorporate additives as methylcellulose or glycerin as anti-washout ingredients, in combination with calcium chloride as an accelerator, to increase the cohesiveness and plasticity of the material, making it easier to handle and accelerating its setting time. [8,14,16-18] Therefore, the aim of this study was to evaluate the influence of 15% glycerin on the physico-mechanical properties of Portland cement including; compressive strength, setting time, cohesion time and anti-washout time in an attempt to apply it to mineral trioxide aggregate (MTA).

## MATERIALS AND METHODS

White Portland cement powder was used in this study and was mixed with 10 wt % calcium chloride ($CaCl_2$) powder as an accelerator, i.e. 0.9 gm white Portland cement powder was mixed with 0.1 gm $CaCl_2$using an electronic balance with four digits precision (Scaltec Instruments GmbH Rudolf-Heiligenstadt, Germany).The formulated powder was mechanically mixed in a vibrator (Jenway 1010 vibrator, Serial #: 2273, Dunmow, Essex, U.K.) for 5 minutes to ensure proper distribution and intermingling of the powder particles. The resultant powder was divided into two groups; in the first group it was mixed with distilled water to form "PC" paste while in the second group it was mixed with 15% glycerin liquid to form "PCG" paste. These two cements "PC" and "PCG" were compared to the commercially available Biodentine (BD).

For the PC group, the powder/ liquid ratio was 3: 1 (according to the manufacturer's recommendations for MTA) i.e. for every 1gm powder, 0.33 ml distilled water was used. On the other hand, for the PCG group, 15% glycerin liquid was mixed with the powder of Portland cement at different weight ratios till attaining an appropriate smooth and homogenous consistency employing the standardized one inch string method. [19] A 2:1 powder/ liquid ratio was selected for the PCG group i.e. for every 1 gm Portland cement powder, 0.5 ml glycerin liquid was used.

The 15% glycerin liquid means that every 100 ml of the liquid is formed of 15 ml glycerin and 85 ml distilled water. This percentage was selected according to a pilot study based on extensive preliminary mixtures and tests. In the pilot study, the criteria used for selecting the appropriate glycerin percentage, to be mixed with Portland cement powder containing10 % $CaCl_2$, was done by evaluating the setting time and compressive strength of each tested sample. For the BD group, Biodentine™ was mixed according to the manufacturer's directions. The materials used in this study are detailed in table (I).

The tested materials (BD, PC, and PCG) were evaluated regarding the following properties:

**1. Compressive strength test:**

Ten specimens of each tested material (BD, PC, and PCG) were prepared according to ISO Standard 9917: 1991 and American National Standards Institute / American Dental Association (ANSI/ADA) Specification No. 96 for water-based dental cements. [20,21]

A cylindrical split Teflon mould of diameter 4 mm and height 8 mm was used for specimens preparation. This mould provided a height to diameter ratio of 2:1. At this ratio, the risk of complicated force distributions as a result of the cone formations overlapping in the ends of the cylinder was reduced and also the risk of specimen buckling was reduced. [21]

The required P/L ratio for each tested material was mixed on a clean dry glass slab. The mixed cement was packed into the mould with a glass slab beneath to ensure a flat surface for condensation. The material was applied in increments and condensed carefully with an amalgam condenser to minimize air entrapment. Moulds were overfilled and the excess material was removed by using a glass slide to ensure properly leveled specimens. After setting, the tops and bottoms of the specimens were ground, until flush with the moulds, using a series of silicon-carbide abrasive sheets; # 800,# 1000 and # 1200 successively to obtain flat and smooth surfaces.

The specimens were removed from the Teflon moulds and examined for voids and irregularities. If voids were detected, the specimen was discarded. The specimens were then stored in distilled water for 24 hours at 37±1°C in an incubator (Foc Incubator, Japan).

At the time of test conduction, each specimen was vertically mounted in a computer controlled universal testing machine (LRX plus, LC 5 KN. Lloyd Instruments Ltd., Fareham, U.K.) with a load-cell of 5 KN and data were recorded using computer software (Nexygen-MT; Lloyd Instruments). Then, each specimen was statically loaded (in a compression manner) along its long axis at a crosshead speed of 0.5 mm/min until failure. The failure load was recorded in Newtons (N). Then, the compressive strength in Mega Pascal (MPa) was calculated using the following formula; [9,20]

$$\delta c = \frac{4\,P}{\pi\, D^2}$$

Where;

$\delta_c$: Compressive Strength in Mega Pascal (MPa)

P: Maximum load at failure in Newtons (N).

π: Constant= 3.14

D: Diameter of specimen in millimeters (4 mm).

**2. Setting time:**

Five specimens of each tested material (BD, PC, and PCG) were prepared according to ISO Standard 9917: 1991 and American National Standards Institute / American Dental Association (ANSI/ADA) Specification No. 96 for water-based dental cements, as well as American Society for

Testing and Materials (ASTM) standard test method for time and setting of hydraulic-cement paste. [20,22]

The required P/L ratio for each tested material was mixed manually on a clean dry glass slab. The mixed cement was packed in a split Teflon mould of diameter 20 ± 1 mm and thickness 1.5 ± 0.1 mm.

The setting time measurement was carried out using an indenter of mass 400 ± 5 gm with a flat end of diameter 1.0 ± 0.01 mm. The flat ended indenter tip is cylindrical for a distance of approximately 5 mm. Three minutes after the start of mixing, the indenter was carefully applied vertically on the surface of the mix and was allowed to remain for 5 seconds to mark definite circular indentation. The indentations were repeated at equal intervals, initially 5 minutes each and then every 1minute interval, till reaching the approximate setting time that was previously estimated in a pilot study, until the indenter tip failed to make a complete circular indentation when viewed using a hand lens of low magnification (X10). The indenter tip was cleaned between indentations.

The time elapsing, between the start of mixing to the time when the needle failed to make complete circular indentation or to penetrate, was recorded as the setting time.

## 3. Observation of cohesion and anti-washout ability:

The cohesion time was measured with a specially designed method described by Chen et al. in 2009 [23] and Liu et al. in 2013 [24] with slight modification to fit the test. Five cylinders from each tested material were prepared using a split metallic mould of diameter 12 ± 1 mm and height 20± 1 mm, with a closed bottom and an opened top for application of the material. The tested cements (BD, PC and PCG) were mixed and immediately packed into the mould from the open side and flattened with a spatula. The whole assembly was vertically immersed in a saline solution for visual inspection of the disintegration of the tested material. To avoid turbidity of the saline solution, the assembly was transferred every three minutes into a new beaker containing fresh saline solution. The cohesion time was calculated starting from the time of immersing the assembly in saline solution.

The cohesion time was defined as the minimum time required to obtain a stable cement paste, i.e. the minimum time when no visible disintegrated particles were observed in saline solution. [25]

Washout refers to the tendency of freshly prepared cement paste to “disintegrate upon early contact with blood or other fluids”. [26] The method used for testing anti-washout ability has been developed by Liu et al. in 2013 [24] and was slightly modified to fit the test. For measurement of anti-washout time, five specimens of each of the tested cements (BD, PC, and PCG) were mixed and packed into a split cubic Teflon mould of 1 cm side length. After three minutes, the specimens were removed from the mould and placed in a saline solution. Each specimen was then hand-scrubbed, while still in the solution, for 20 seconds at 1 minute intervals. The specimen was considered washout-proof and the corresponding time was recorded when there was no visible surface abrasion on the tested cement specimen after scrubbing. [27]

## 4. Scanning Electron Microscopy (SEM):

SEM was used for internal microstructural evaluation of the three tested cements (BD, PC, and PCG).One representative specimen from each material was prepared using the moulds of the anti-washout ability test. The specimens were left to dry at room temperature for 24 hours.

To analyze the internal microstructure, the specimens were sectioned into two halves using a disposable surgical scalpel blade # 15 to initiate the crack. The fractured surfaces of the specimens were sputter-coated with gold to render the surface electrical conductivity. Each specimen was mounted on an aluminum stub with the fractured surface facing upwards. Then, the specimens were examined by SEM (Model Quanta 250 Field Emission Gun "FEG" with accelerating voltage 30 KV. FEI Company, Netherlands) at 1000x magnification.

## Statistical Analysis

Data management and analysis were performed using Statistical Package for Social Sciences (SPSS) vs. 17. Comparisons between groups were done using the Kruskal-Wallis test followed by the post hoc Bonferroni test. [28] All p-values are two-sided. P-values ≤0.05 were considered significant.

**TABLE (I): The chemical composition and the manufacturers of the materials used in this study.**

| Material | Composition | Manufacturer | Lot # |
|---|---|---|---|
| Biodentine™ | Powder:<br>Tri-calcium Silicate<br>Di-calcium Silicate<br>Calcium Carbonate and Oxide<br>Iron Oxide<br>Zirconium Oxide<br>Liquid:<br>Calcium chloride ($CaCl_2$)<br>Hydrosoluble polymer | Septodont, SaintMaur - des-Fossés, Cedex, France | B02380 |
| White Portland Cement | Powder:<br>Tri-calcium silicate<br>Di-calcium silicate<br>Tri-calcium aluminate<br>Calcium sulfate dihydrate<br>Liquid:<br>Distilled water | Helwan cement company, Helwan, Cairo, Egypt | |
| Calcium chloride ($CaCl_2$) | Calcium chloride powder | Sigma-Aldrich, Inc, St Louis, MO, USA. | 40388 |
| Glycerin ($C_3H_8O_3$) | Odorless colorless propanetriol liquid (Propane -1,2,3- triol) | Sigma-Aldrich, Inc, St Louis, MO, USA. | L33G202 |

# RESULTS

**Table (II): The means and standard deviations (S.D.) of the tested properties for each investigated material.**

| Material | BD | | PC | | PCG | | |
|---|---|---|---|---|---|---|---|
| Property | Mean | S.D. | Mean | S.D. | Mean | S.D. | P-value |
| Compressive Strength (MPA) | 59.3a | 2.6 | 17.2c | 2.6 | 48.0b | 7.2 | <0.001 |
| Setting time (min) | 24.0b | 2.4 | 57.0a | 6.5 | 18.0b | 6.4 | 0.005 |
| Cohesion time (min) | 7.2b | 1.3 | 24.2a | 5.1 | 4.2b | 1.6 | 0.003 |
| Anti-washout time (min) | 10.2b | 1.5 | 33.2a | 6.1 | 7.0c | 1.2 | 0.002 |

Group means sharing same letters are not significantly different from each other, p-value>0.05.

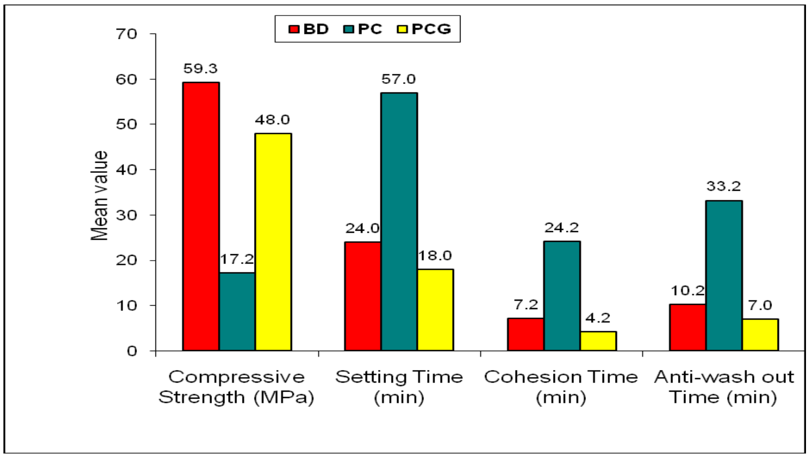


**Figure (1): Mean values of the four tested properties for each investigated material.**

Table II and figure 1 present the mean values of compressive strength, setting time, cohesion time and anti-washout time of the three investigated materials (BD, PC and PCG).

BD provided the highest compressive strength (59.3 MPa ± 2.6) followed by PCG (48 MPa ± 7.2) while the lowest compressive strength was provided by PC (17.2 MPa ± 2.6). The highest setting and cohesion times were exhibited by PC (57 min ± 6.5 and 24.2 min ± 5.1 respectively) while there was no significant difference between BD and PCG. Regarding the anti-washout time, PC exhibited the highest value (33.2 min ± 6.1) followed by BD (10.2 min± 1.5) while the lowest value was provided by PCG (7 min ± 1.2).

**Scanning Electron Microscopy (SEM):**
Figures 2-4 show the internal microstructure of the three investigated materials (BD, PC and PCG).

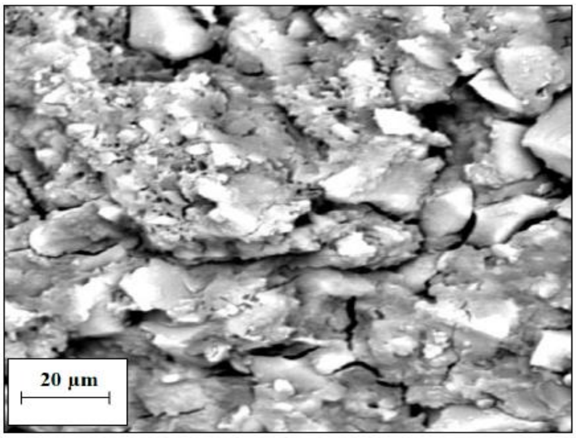


**Figure (2): SEM photomicrograph of thefractured surface of BD at 1000x, demonstrating block-like crystals with minimal porosities.**

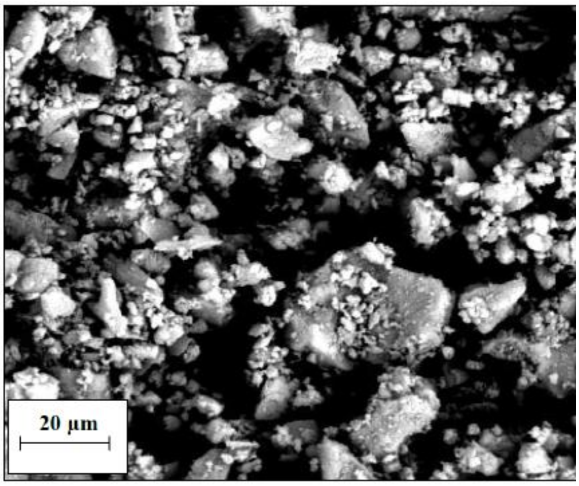


**Figure (3): SEM photomicrograph of thefractured surface of PC at 1000x, demonstrating lack of cohesion between the particles with excessive porosities.**

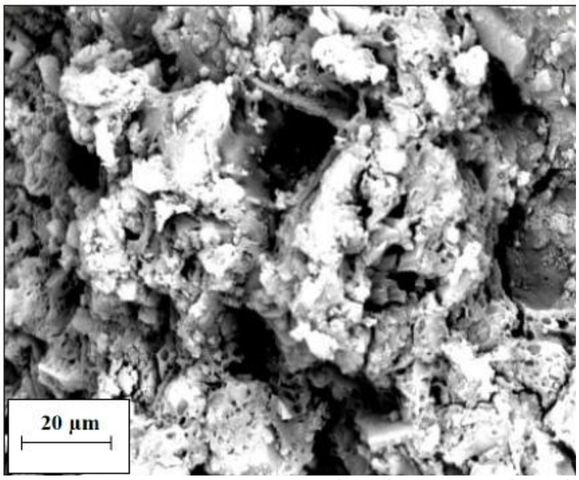


**Figure (4): SEM photomicrograph of the fractured surface of PCG at 1000x, demonstrating globular coherent glue-like crystals with few porosities.**

## DISCUSSION

Portland cement contains the same principal chemical elements of MTA. [9] Modifications of Portland cement can be used to aid in further development of MTA, aiming to improve its physical characteristics and expand its scope of clinical applications.

The main disadvantages when using Portland cement or MTA are their extended setting time and difficult handling. Their normal setting time ranges from 3 to 4 hours. [1,4,9,15] The setting time of dental cements is an inherent property depending on the rate of matrix formation. It refers to the period during which matrix formation has reached a point at which an external physical disturbance will not cause permanent dimensional changes. [8]

Calcium chloride ($CaCl_2$) stimulates the hardening process of MTA and Portland cement. Thus, it is one of the commonly used cement accelerators. It provided significant reduction of setting time without significantly affecting other physical properties. [8,17,29] At the same time, $CaCl_2$ offers a proper consistency of Portland cement with low water/powder ratio. [29]

Portland cement and MTA are hydraulic cements that can set and harden under water through the hydration of the component cement minerals. [9] The setting of the cement is mainly attributed to the formation of a solid network, which is related to the hydration rate. Therefore, the acceleration of hydration will result in a decrease in the setting time. [13,15,30] The mechanism of action of $CaCl_2$ is not fully understood, but it is believed that $CaCl_2$ is partially consumed during the hydration process by reacting with tricalcium aluminate, forming chloroaluminate. [8] Several previous studies reported that addition of 10% $CaCl_2$ was effective in reducing the setting time of Portland cement. [13,31,32] Moreover, *Harrington in 2005,* stated that addition of 10 to 15% calcium chloride ($CaCl_2$) to Portland cement decreases its setting time more than 50%. [33]

Therefore, it was decided in this research to incorporate 10 wt % $CaCl_2$ in the Portland cement powder of both PC and PCG groups to increase the rate of hydration of the cement thereby accelerating the setting time. Moreover, $CaCl_2$ was added to simulate the composition of the commercially available BD as a means of standardization.

Moreover, the accelerating effect of $CaCl_2$ is evident when comparing the setting time of BD and PC in this study (24 and 57 min respectively) with the setting time of Portland cement in previous studies (3-4 hours). However, BD is characterized by obviously short setting time (24 min) which is significantly lower than PC (57 min). This may be attributed to decreased particles size as well as reduced water content of the BD system which further reduced the setting time and increased the compressive strength. The reduced water content was made possible by the addition of a water soluble polymer (softener) as well as a new pre-dosed capsule formulation for use in a mixing device. The modified formulation of Biodentine has largely improved its physical properties (as shown in fig. 2), making it much more user-friendly. [10,14] Most of the data available on Biodentine is forthcoming from the manufacturer with very few independent researches being conducted. [10]

In industry the setting time of Portland cement can be controlled by the use of a superplasticizing admixture to make the material more workable and to obtain a homogeneous flowable mix. [16] Superplasticizers are very effective dispersing agents that reduce the water content of the mix while retaining its workability. This results in an increase in compressive strength and significant reduction in setting time, which reduces the risk of dislodgement and contamination. [16,17]

The commonly used superplasticizers are those tested by *Papo and Piani in 2004,* [34] *Camilleri et al. in 2005,* [16] *Camilleri in 2008,* [17] and *Wongkornchaowalit and Lertchirakarn in 2011,* [14] which are either accelerating agents based on a modified polyacrylic resin or retarding agents based on polycyclic copolymers with modified structures, carrying hydroxylated side chains. Moreover, the United States Patent Number 5,340,385 by *Arfaei et al. in 1994* for hydraulic cements stated that the incorporation of glycerol, as a set-accelerating admixture, was found to be surprisingly effective in decreasing the setting time of hydraulic cements. [35]

Accordingly, it was indicated that with a fixed hydraulic cement particle size, the most suitable and applicable strategy to improve the handling characteristics of the cement is to increase the viscosity of the mixing liquid. This is the only method that can improve the handling characteristics of the cement without compromising either the setting time or the mechanical properties, which are also crucial properties for successful clinical application of Portland cements. [10,14,24,36]

Meanwhile, glycerin (glycerol) was added to Portland cement in the present study in an attempt to improve its handling properties. Glycerin is a colorless, odorless, sweet-tasting and viscous liquid that is widely used in pharmaceutical formulations. [37] It is a simple polyol compound with multiple hydroxylfunctional groups available for organic reactions. Glycerin has three hydroxyl groups that are responsible for its solubility in water and its hygroscopic nature. [18,37] Hygroscopy is the ability of a substance to attract and hold water molecules from the surrounding environment through either absorption or adsorption. Consequently, water molecules become suspended between the material's molecules which somewhat changes the material physically by an increase in volume and stickiness. [10,37]

By analyzing the results of this study, it can be seen that Portland cement with 15% glycerin (PCG) showed significant decrease in setting, cohesion and anti-washout times (18 min, 4.2 min and 7 min respectively) when compared to Portland cement "PC" (57 min, 24.2 min and 33.2 min respectively). On the other hand, there was no significant difference between PCG and BD groups for both setting and cohesion times, while there was a significant difference between both groups for anti-washout time test.

Being a viscous liquid, glycerin may have increased the inter-particle interaction of the Portland cement resulting in improvement in its cohesiveness and homogeneity (see fig 3 and 4). Thismay have effectively reduced the washout ability of the cement which significantly overcomes its initial looseness. Moreover, glycerin is a set-accelerating admixture as demonstrated by *Arfaei et al. in 1994.* [35]

In fact, the cohesion of a Portland cement paste can be viewed as a competition between forces acting on Portland cement particles and forces acting between the Portland cement paste and the surrounding fluid. The latter forces are mainly governed by the difference in osmotic pressure between the cement interstitial fluid and the surrounding liquid. [24] Glycerol molecules might have been adsorbed onto Portland cement particles and their polymer chains might have intertwined and entangled which might have resulted in strong forces between particles, improving the cohesion of the cement. Moreover, the high viscosity of the polymer solution can prohibit the migration and release of Portland cement particles into the surrounding fluid, improving the anti-washout ability. This was in accordance with other studies. [10,24]

PCG exhibited shorter cohesion and anti-washout times than BD which may be attributed to the presence of the water-soluble polymer in Biodentine composition. This water-soluble polymer has a surfactant effect and thus it may disperse the cement particles by applying a charge on their surfaces. [10] This dispersion may lead to a fluid mixture which might have resulted in dislodgement of the Biodentine when tested for washout. These findings were in agreement with Grecha et al. in 2013. [10]

It is well known that, from a material science point of view, the mechanical properties of a material are determined by its microstructure. Investigating mechanical properties without relating them to microstructure is impractical and meaningless for both theoretical understanding and effective designing for targeted properties. [24]

Regarding the compressive strength of Portland cements, it is not solely an intrinsic property of the material, but it also depends both on the shape and size of defects. [24] It is worth noticing that the strength of a material generally decreases with increased porosity. According to SEM microstructural findings of this study (Fig 4), glycerin has improved the cohesion between the particles, thereby reducing the porosities. Thus, it can be concluded that glycerin also has a strengthening effect on Portland cement. This is supported by the results of this study, as PCG provided a significant increase in the compressive strength (48 MPa) compared to PC (17.2 MPa). Similarly, the microstructural features of BD showed block-like crystals with minimal porosities (Figure 2) which was in accordance with the significantly high results obtained from compressive testing (59.3 MPa). On the other hand, PC showed the lowest compressive strength (17.2 MPa) which is supported by figure 3that shows lack of cohesion between the particles with excessive porosities. Most of the pores are formed by the remaining aqueous solution after the hydrolysis of the initial inorganic compounds.

Finally, incorporation of 15% glycerin into the Portland cement accomplished two things. First, it increased the cohesiveness and plasticity (or moldability) of the material, making it easier to handle. Second, it increased the washout resistance, a benefit when placed in a contaminated (wet) site. Moreover, it increased the viscosity of the mix and provided rapid setting. Without controlling the particle size and shape, PCG has provided satisfactory results in comparison to BD that has reduced particle size. Modification of the particle size and shape of PCG may further improve its properties.

It is worth noticing that addition of 15% glycerin may have a positive effect on the fracture behavior, rendering Portland cement with a kind of tolerance to damage. This tolerance to damage is very interesting for biological applications. The material, in the case of an overload, will nevertheless retain some degree of mechanical integrity, this will on one hand prevent debris from escaping the implant site, and on the other hand allow the material to be able to withstand further loading. [24]

## CONCLUSION

Based on the results of this study, it can be concluded that addition of 15% glycerin to white Portland cement has improved the setting time, the cohesiveness and the anti-washout ability of the cement with subsequent improvement in the handling properties as well as increasing its compressive strength. This trial is considered to be a successful step which should be performed on Portland cement twin; the MTA.

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