



EVALUATION OF COMPRESSIVE STRENGTH AND SETTING TIME OF SOME TYPES OF CALCIUM SILICATE-BASED MATERIALS MODIFIED WITH CHICKEN EGGSHELL POWDER

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ABSTRACT

Objective: This study evaluated compressive strength and setting time of some types of calcium silicate-based materials modified with chicken eggshell powder (CESP). **Material And Methods:** The samples were classified into eight major categories, including Group 1 (non-modified glass ionomer cement (GIC) as a control group), Group 2 (1 wt.% eggshell incorporated GIC powder), Group 3 (3 wt.% eggshell incorporated GIC powder), Group 4 (5 wt.% eggshell incorporated GIC powder), Group 5 (non-modified biodentine (BD) as a control group), Group 6 (1 wt.% eggshell incorporated (BD) powder) Group 7 (3 wt.% eggshell incorporated (BD) powder) and Group 8 (5 wt.% eggshell incorporated (BD) powder). **Results:** (group 2), (group 6) exhibited the highest statistically significant compressive strength means values, while (group 4), (group 8) exhibited the lowest statistically significant setting time means values, **Conclusion:** Addition of CESP to GIC and BD can increase compressive strength and decrease setting time of materials.

KEYWORDS: Compressive strength, biodentine, eggshell powder, setting time, glass ionomer

INTRODUCTION

Calcium Silicate based Materials (CSMs) have several indications of use in endodontics and their potential clinical applications have gained popularity in recent years. The CSMs seem to have essential properties tailored for their clinical use such as the good sealing correlated to expansion, the ability to set in the presence of fluids, the release of ions acting as epigenetic signals and bioactivity⁽¹⁾. Some bioactive dental materials include calcium hydroxide cements, glass ionomer cement, and other

newer tricalcium silicate based cements (TCS) such as biodentine⁽²⁾.

Glass-ionomer cement (GIC) is considered to be the most important tool in the fighting against the development and prevention of dental caries it acts as a reservoir of other ions including fluoride in the oral environment and a mechanical barrier between the surface of tooth and bacteria protecting the tooth surface⁽³⁾. The most important property of GIC is that it can provide the most prominent seal under the most challenging clinical conditions in the oral

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cavity. Clinically, GICs are excellent restorative material of choice for the posterior teeth in primary dentition ⁽⁴⁾ .

Although GIC is regularly used as a desirable restorative material in dentistry, they have disadvantages too, lack of sufficient strength, lack of toughness, technique-sensitive, poor wear resistant, resistance to fracture is poor, unaesthetic few shades, low bond strength and compressive strength ⁽⁵⁾ .

A new bioactive cement, also called as Smart Dentin Replacement (SDR) and popularly known as biodentine (BD), was recently launched as a dentine substitute. It shares both its indications and mode of action with calcium hydroxide but does not have its drawbacks. This new calcium silicate-based material exhibits physical and chemical properties similar to those described for certain Portland cement derivatives ⁽⁶⁾ .

On the biological level, it is perfectly biocompatible and capable of inducing the apposition of reactionary dentin by stimulating odontoblast activity and reparative dentin by induction of cell differentiation ⁽⁷⁾ . It is in effect a dentine substitute that can be used as a coronal restoration material (for indirect pulp capping) but can also be placed in contact with the pulp. It's faster setting time allows either immediate crown restoration or to make it directly intraoral "functional" without fear of the material deteriorating ⁽⁸⁾ .

Biodentine does not seem to satisfy the requirements of most clinicians with regard to setting time ⁽⁶⁾, radiographic detectability, compressive strength ⁽⁹⁾ and the absence of the caries inhibiting materials ⁽¹⁰⁾. As pointed out previously, calcium silicate-based materials could be improved by having caries inhibition capacity, improved chemo-physical, mechanical and bioactivity. One way of achieving this could be via incorporation of eggshell powder which have been used extensively in other dental applications.

Egg shell has recently been described as a rich source of minerals containing calcium, phosphorus, magnesium and strontium ⁽¹¹⁾. Egg shell being a rich source of calcium , contains 94% calcium carbonate, 1% calcium phosphate, 1% magnesium carbonate and 4% organic matter ⁽¹²⁾.

So, hypothesis of this study was done that there will be an effect on evaluation of compressive strength and setting time of eggshell modified calcium silicate-based cement.

MATERIAL AND METHODS

The materials for this investigation were glass ionomer filling material (FETIHMH.MAHIR 42030 KONYA TURKEY), Biodentine (SEPTODONT, SAINT MAUR DES FOSSÉS, FRANCE) and Eggshell powder (ESP).

Grouping of samples:

This study evaluated a total of 192 samples. The samples were classified into eight major groups (n=24) based on their alterations:

Group 1: unmodified GIC.

Group 2: GIC modified with 1% weight ESP.

Group 3: GIC modified with 3% weight ESP.

Group 4: GIC modified with 5% weight ESP.

Group 5: unmodified BD.

Group 6: BD modified with 1% weight ESP.

Group 7: BD modified with 3% weight ESP.

Group 8: BD modified with 5% weight ESP

Each main group was then divided into two subgroups (n= 12) based on the type of test used: compressive strength and setting time test.

Preparation of eggshell powder

Eggshell employed in this study was prepared in Department of Chemistry, Faculty of Science, Boys, Cairo, Al-Azhar University by the process

of calcination following the protocol given by world property intellectual organization (WO/2004/105912: Method of producing eggshell powder). Eggshells were cleaned in distilled water. The eggshells then kept in hot water bath at 100°C for 10 minutes followed by removing the membrane. These eggshells then crushed using a sterile mortar and pestle. The crushed particles then heated at 1200°C in a muffle furnace and powdered to small particles. ⁽¹³⁾

Samples preparation for glass ionomer

Glass ionomer samples were prepared in Department of Dental Biomaterials, Faculty of Dental Medicine, Boys, Cairo, Al- Azhar University according to the manufacturer's instruction. Use the accompanying measuring spoon to measure exactly one level scoop of powder and place it on the mixing pad or according to need. Carefully shake the liquid and dispense one drop. Mix the two by gently folding the powder into the liquid; mixing should take up to 15 seconds. Modification of glass ionomer was prepared by adding 1%, 3%, and 5% by weight of eggshell to glass ionomer powder. If we weigh from glass ionomer powder 0.7 gm, the weight of eggshell added to the glass ionomer powder was calculated using the following equation $(0.7 \times w) / 100$. Eggshell modifier was added to the glass ionomer powder and placed in capsule then mixed it in the amalgamator (HL-AH G5 Dental Digital Amalgamator) for 10 seconds. This was to ensure dispersion of eggshell into the glass ionomer powder as extensively as possible. Eggshell modified glass ionomer was mixed as mentioned above.

Samples preparation for biodentine

Biodentine samples were prepared in Department of Dental Biomaterials, Faculty of Dental Medicine, Boys, Cairo, Al- Azhar University according to the manufacturer's instruction by adding five drops of the liquid to the powder and triturating in amalgamator for 30 seconds at 3000 rpm leading

to the formation of a paste of creamy consistency. Modification of biodentine was prepared by adding 1%, 3%, and 5% by weight of eggshell to biodentine powder. Since biodentine powder weight is 0.7 gm. So, the weight of eggshell added to the biodentine powder was calculated using the following equation $= (0.7 \times w) / 100$. Eggshell modifier was added to the biodentine powder and mixed it at 3000 rpm in the amalgamator for 10 seconds. This was to ensure dispersion of eggshell into the biodentine powder as extensively as possible. Five drops of liquid added to the powder and triturating in amalgamator for 30 seconds at 3000 rpm leading to the formation of a paste of creamy consistency.

Compressive strength test:

Each group's samples were made by mixing the respective powder with the liquid per the manufacturer's guidelines. The mixed material from each group was condensed in split molds (3mm diameter x 6mm height) material was poured into the mold and covered with a glass slide until the initial setting occurred. Following solidification, prior to the experiment, samples (n=12 per group) were stored in distilled water at 37°C for 24 hours.

All samples were individually and vertically mounted on a computer controlled universal testing machine with a load cell of 5 KN and data were recorded using computer software. The samples were statically loaded (in compression manner) using stainless-steel rod ended with flat plate (40mm x 60mm) attached to the upper movable compartment of the machine at a cross head speed of 1 mm/min until failure. The maximum failure load was recorded in N and converted into MPa. The compressive strength was calculated from the recorded peak load divided by sample surface according to the following equation.

Compressive strength = $4P / \pi d^2$ Where P is the maximum load applied in Newton and d is the diameter of the sample in millimeters.

Setting time test:

Samples of all materials (glass ionomer and biodentine) were prepared according to ANSI/ADA specification No. 96 for water-based dental cements. ⁽¹⁴⁾ Each sample was prepared by mixing the powder with liquid according to manufacture instruction. Then, mix was packed in a specially designed Teflon mold 8mm x 10mm x 5mm in dimension. The stopwatch was started when the liquid is added to the cement. Recorded this time (T1). The mold was filled by cement resting on a glass slap with the cement paste gauged. The mold was filled completely and smooth off the surface of the paste making it level with the top of the mold. Then, the mold was placed and rested on the glass slap, under the rod bearing the needle. The needle was lowered gently until it comes in contact with the surface of cement and quick release, allowing it to penetrate into cement. In the beginning, the needle completely penetrated the cement and was repeated every 2 minutes till the needle failed to penetrate the cement for about 0.5 mm measured from the bottom of the mold. (T2). For determining the final setting time, replace the needle of the Vicat's apparatus by the needle with an annular attachment. The cement was considered finally set when upon applying the final setting needle gently to the surface of the cement; the needle was made an impression on the surface of the cement, while the attachment fails to do so. Record this time (T3).

Initial setting time=T2-T1

Final setting time=T3-T1

Statistical Analysis

The compressive strength and setting time of groups were compared using a one-way analysis of variance (ANOVA). The F test is utilized to compare paired means between test groups in all analyses. The computation is carried out using the software PASW Statistics 17 (SPSS Inc., Chicago, IL, USA), with all reach a certain threshold to an accuracy of 0.05.

RESULT

Compressive strength of glass ionomer cement:

The statistical analysis results showed that; the highest (mean \pm SD) value of compressive strength was recorded for GIC modified by 1wt. % ES group (21.84 \pm 0.604 MPa) followed by 3wt. % ES group (15.493 \pm 1.422 MPa), and 5wt. % ES group (10.34 \pm 1.665 MPa) respectively. The lowest (mean \pm SD) value of compressive strength was recorded for unmodified GIC (4.58 \pm 0.097 MPa).

Among the groups, Tukey's pair-wise post-hoc test showed statistically *significant* difference ($p < 0.05$) between all tested groups as seen in figure (1)

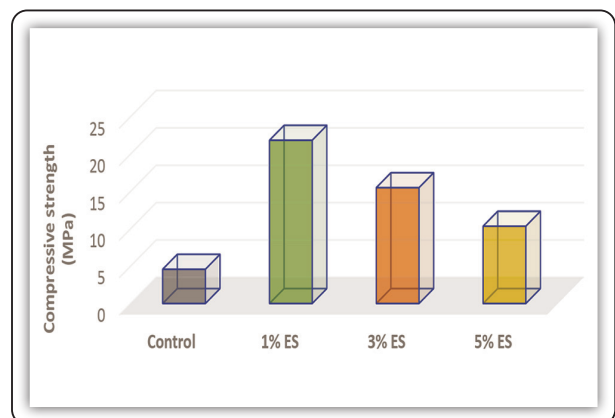


FIG (1) Column chart representing means of compressive strength for glass ionomer groups.

Compressive strength for biodentine:

The statistical analysis results showed that; the highest (mean \pm SD) value of compressive strength was recorded for biodentine modified by 1wt. % ES group (67.97 \pm 1.954 MPa) followed by 3wt. % ES group (38.90 \pm 4.164 MPa), and 5wt. % ES group (25.58 \pm 1.095 MPa), respectively. The lowest (mean \pm SD) value of compressive strength was recorded for unmodified biodentine (22.58 \pm 1.176 MPa). Among the groups, Tukey's pair-wise post-hoc test showed statistically *significant* difference ($p < 0.05$) between all tested groups as seen in figure (2).

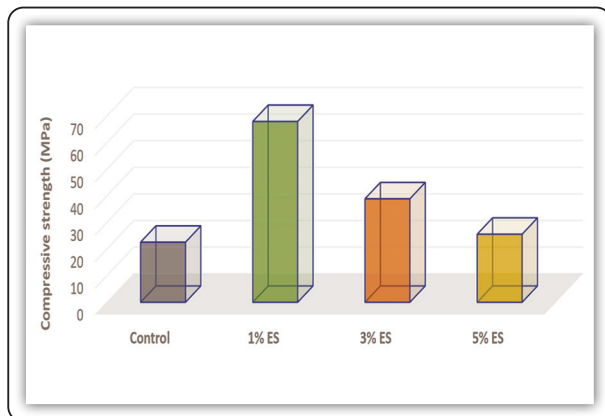


FIG (2) Column chart representing means of compressive strength for biodentine groups.

Setting time:

Setting time for glass ionomer cement

The statistical analysis results showed that; the highest (mean \pm SD) value of setting time was recorded for unmodified GIC (12.596 \pm 0.376 min) followed by GIC modified by 1wt. % ES group (10.066 \pm 0.647 min), and 3wt. % ES group (9.035 \pm 0.802 min) respectively. The lowest (mean \pm SD) value of setting time was recorded for 5wt. % ES group (8.533 \pm 0.449 min).

Among the groups, Tukey's pair-wise post-hoc test showed statistically *significant* difference ($p < 0.05$) between all tested groups except for GIC modified with 3wt.% ES and 5wt.% ES groups there was no statistically significant difference ($p = 0.47572$) as seen in figure (3).

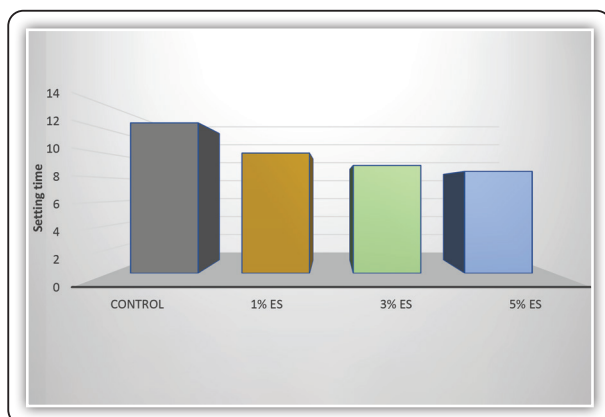


FIG (3) Column chart representing means of setting time for glass ionomer groups.

Setting time for biodentine

The statistical analysis results showed that; the highest (mean \pm SD) value of setting time was recorded for unmodified biodentine (24.85 \pm 1.01 min) followed by biodentine modified by 1wt. % ES group (19.72 \pm 0.788 min), and 3wt. % ES group (14.05 \pm 1.283 min) respectively. The lowest (mean \pm SD) value of setting time was recorded for 5wt. % ES group (11.535 \pm 0.427 min) as seen in figure (4).

Among the groups, Tukey's pair-wise post-hoc test showed statistically *significant* difference ($p < 0.05$) between all tested groups.

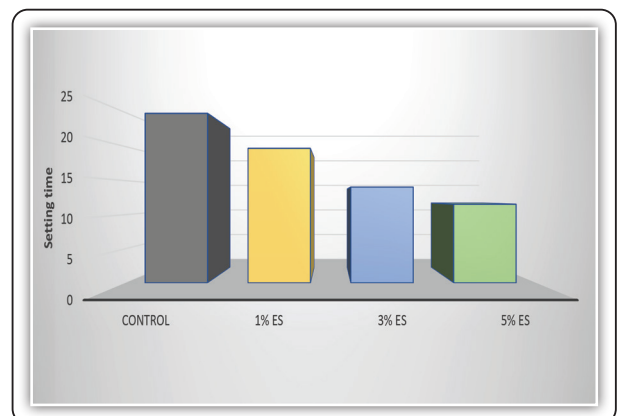


FIG (4) Column chart representing means of setting time for biodentine groups.

DISCUSSION

Calcium silicate-based materials have undergone various improvements since its introduction, which in turn led to better characteristics, such as increased strength, improved handling characteristics, and enhanced wear resistance⁽¹⁵⁾. On the other hand, increasing worldwide interest in sustainable technologies led to the invention of products with lower impact on the environment⁽¹⁶⁾.

Eggshell is one of the by-products of households, restaurants, and food industries, which is daily produced in massive amounts and has been categorized as one of the worst environmental problems worldwide due to its chemical composition and availability. At the same time, eggshell is

considered the best natural source of calcium⁽¹⁷⁾. Thus, the present study sought to test the effect of adding eggshell powder to the powder component of CSMs on some of its mechanical and physical properties.

The results of the present study showed that, eggshell modified calcium silicate based materials (glass ionomer and biodentine) have high compressive strength than unmodified groups. These results are in agreement with those of *Lubis et al.*⁽¹⁸⁾ who also concluded that eggshell filler added to acrylic resin used in the construction of denture base not only improved the mechanical properties, but also provided a cost-effective and renewable filler material that could be used instead of more costly alternatives.

Also, *Rahmi et al.*⁽¹⁹⁾ deduced that the incorporation of eggshell particles in cross-linked composites enhances its mechanical properties; they also suggested that the utilization of this novel filler material can lead to the production of low-cost polymer composites.

This may be due to load transfer between the filler with the matrix as a result of adhesion. Also, the addition of filler causes the increasing of threshold stress point from material since fillers absorb some of the load that received by it, so it can increase the strength of material.⁽²⁰⁾

Also, it was found that 1% eggshell modified calcium silicate-based material show high strength followed by 3% then 5%. This mean that strength of material decreases as concentration of eggshell modifier increase. This may be attributed to that, increasing of filler, while the number of matrix was decrease, so it cause a narrow area between phases where the filler is not evenly distributed in the matrix which cause the formation of empty fraction in the material, also it may be due to that , if the concentration of filler increase too much, so it causes accumulation of filler particle which effects in the uneven distribution of filler particle⁽²¹⁾.

The result of the present study showed that eggshell modified calcium silicate-based materials have short setting time than unmodified groups. This result are in agreement with *Mtallib* and *Rabiu*⁽²²⁾, *Lothenbach et al.*⁽²³⁾ as they investigated the effect of eggshell on the setting time of cement and concluded that the higher the eggshell content, the faster the setting time rate.

This may be due to that, using CaCO₃ as a filler can accelerate the cement hydration, especially calcium silicate, which is the major component of cement and provides a nucleation site for the hydration products to precipitate. The presence of CaCO₃ in cement produces calcium carboaluminates when calcium aluminate reacts with the calcium carbonate (CaCO₃). The advantage of using filler, like CaCO₃, can lead to low capillary porosity and might also increase early strength and finally decreased the setting time⁽²⁴⁾.

CONCLUSION

Within the limitation of this study, it can be concluded that, the addition of ESP to GIC and BD can increase compressive strength and decrease setting time.

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