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Mechanical Properties of New Cement-Based Capping Material Prepared from Egg Shell and Chitosan

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Abstract: The aim of this study was to evaluate the compressive strength of newly prepared cement-based capping material and biopolymer (Chitosan). The cement was prepared from egg shells and a biopolymer (chitosan), to provide compressive strength. Thirty samples were prepared and their dimensions according to American Dental Association (ADA) specification No.30 were 6 mm in height and 4 mm in diameter. Ten experimental cement samples, 10 glass ionomer cement samples and 10 biodent in samples were used. The compressive strength of the formula of 1spoon of experimental powder with 8 drops of the solution and 1drop chitosan with 8 drops polyphosphonoic acid was 25.60±0.515 MPa which was higher than that of the other formulae. Within the limitations of the experimental methods used in the present study, the cement-based capping material prepared from egg shells and the biopolymer chitosan shows an acceptable compressive strength property.

INTRODUCTION

Calcium-based cements are currently used in dentistry for direct or indirect pulp capping, apexification, apexogenesis and root canal filling. They have certain advantages such as antimicrobial and anti-inflammatory activities. An important requirement for operative and preventive dentistry is the development of restorative materials that can induce remineralization of hypomineralized carious dentin (demineralized/carious dentin). Currently, no restorative materials with proven capability to induce dentin remineralization are available on the market[1]. Compressive strength testing is the most commonly used method to evaluate the strength of these materials[2]. Egg shells are waste materials from hatcheries, homes and the fast food industry and a large supply can be readily obtained.

The egg shell composition is ~98.2% calcium carbonate 0.9% magnesium and 0.9% phosphate[3, 4]. Thus, the egg shell is a rich source of mineral salts, mainly calcium carbonate. Eggshell calcium is probably the best natural source of calcium and it is a better calcium source than limestone or coral sources[3].

Chitin, the precursor to chitosan was first discovered in mushrooms by the French professor, Henri Braconnote in 1811. Chitosan has various biological activities. In dentistry and related areas, the anti-inflammatory reaction, the acceleration of wound healing processes in both the

soft and hard tissues and the antibacterial effect (the bacteriostatic and/or the bactericidal actions)of chitin were mainly investigated to determine its relationship to clinical use. Additionally, chitosan is also applied as the scaffold and as a carrier for molecular therapies, such as drug and the gene delivery systems. These biomedical applications are completed through interdisciplinary collaboration[5, 6]. The aim of this study was to prepare a new cement-based capping material using egg shells and biopolymer chitosan and to evaluate the material's mechanical properties through a compressive strength test.

MATERIALS AND METHODS

Preparation of the tested material

The powder: The powder of the material has several components. Calcium oxide is obtained from chicken egg shells. The egg shell is cleaned using tap water and then the internal protein layer is removed from the shell. The shell is then crushed and heated to 900°C for 1 h in a furnace (Manfredi, Italy). At this temperature, the shell becomes porous, fragile and very white in color. The egg shell $CaCO_3$ decomposes during this decarbonation process and results in CaO and CO_2, according to the reaction[7]:

$$CaCO_3 \rightarrow CaO + CO_2$$

One hundred grams of egg shell provides 56 g of CaO. Hydroxy appetite (Chemical point/Germany), magnesium oxide (Chemical point/Germany), bismuth oxide (Chemical point/Germany) and calcium acetate (Chemical point/Germany) are the other components.

Polyphosphonoic acid solution: The polyphosphonoic acid solution consists of 52% vinyl phosphonic acid, iatonic acid 2:1 and 5% malic acid[8].

Chitosan solution: The chitosan solution (medium molecular weight) was purchased from (Sigma Aldrich, USA). This solution was made by dissolving 1 g of chitosan powder in 100 mL of acetic acid (0.1 mol L^{-1}). The solution was then by stirred and heated at about 55°C in an oven overnight to form a clear homogenous 10 g L^{-1} chitosan solution[9].

The powder of the test material was composed mainly of calcium oxide (70%) magnesium oxide (25%), hydroxy appetite (3%), calcium acetate (0.5%) and bismuth oxide (1.5%). The powder particle size was standardized using a 25 μm sieve and the mixture was mixed using a grinder[10].

The powder-to-liquid ratio was determined by trial using 1spoon powder/8 drops liquidand1spoon powder/10 drops liquid. For all liquid formulae, 1 drop chitosan/8 drops polyphosphonoic acid, 1drop chitosan/10 drops polyphosphonoic acid and 1drop chitosan/20 drops polyphosphonoic acid were combined and the setting time and compressive strength test were used to evaluate the compound. The ratio of 1spoon powder/8 drops liquid for the formula to 1 drop chitosan/8 drops polyphosphonoic acid was the best ratio that provided an acceptable working time and the hardest compound compared with the other formulae. Thus, this ratio was used in this study.

Mixing, working, and setting time

Mixing time: The mixing time was defined as the portion of the working time required to obtain a satisfactory mix of the components.

Working time: The working time was defined as the period of time, measured from the start of mixing, during which it is possible to manipulate a dental material without adversely affecting its properties.

Setting time: The setting time was defined as the period of time measured from the end of mixing until the material had set. The setting time of the material was measured under a controlled temperature and humidity (37°C±1°C and 95±5% relative humidity). The materials were mixed and inserted into the metallic ring molds (10 mm diameter and 2 mm thick), according to the ANSI/ADA Specification No. 30 for temporary restoration bases. Five specimens were made and 180 sec after the end of mixing, the indent or was lowered vertically onto the surface of the cement and allowed to remain there for 15 sec. Each specimen was indented using a standard indentation needle (made in our laboratory), using a 400-gindentation needle with a tip diameter of 1mm to determine the initial setting time.

The setting time was tested for 30 experimental material formulae with a total of five specimens per material formula.

This was repeated at half-minute intervals. The setting time is the number of minutes elapsed from the start of the mixing to the time when the needle fails to make a perceptible circle on the surface of the specimen when viewed by the naked eye. The needle was cleaned between indentations. The setting time was reported to the nearest minute[10]. The mixing time was 45 sec., working time was 2 min and setting time was 5.45 min.

Compressive strength test: Compressive strength is the maximal stress required to fracture a structure. It is considered to be important in restorative dental materials, and this test is the most commonly used method to evaluate the strength of these materials. Specimen dimensions were 6×4 mm, based on ANSI/ADA Specification No. 30 for temporary restoration bases[11]. A metallic mold was made (6 mm in height and 4 mm in

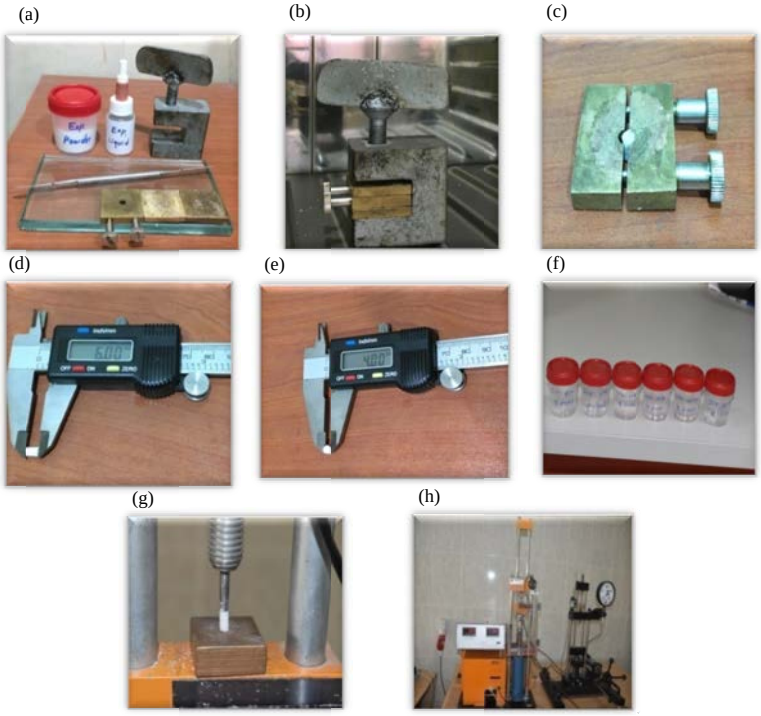

Fig. 1(a-h): The compressive strength test procedure (a) Materials and devices used, (b) Metal mold and screw clamp in incubator, (c) Specimen separation from the mold (d) The length of the specimen, (e) The diameter of the specimen, (f) Specimens in the plastic container, (g) Testing of the specimen and (h) Testing machine

diameter) and the top and bottom metal plates were used. For each group, 10 specimens were fabricated, for a total of 30 specimens. Group I included10 specimens for the cement-based capping material experimental group, group II included 10 specimens for the glass ionomer cement control group and group III included 10 specimens for the biodent in control group. The metallic mold was previously isolated using Vaseline. The material was mixed on a glass slab with a cement spatula for 45 sec and packed to a slight excess into the split mold which was placed on the bottom plate within 1 min of completing the mixing. The top plate was then placed into position and squeezed together with some pressure and any extruded cement was removed. The mold and plates were placed into a screw clamp and screwed together tightly, within 2 min completing the mixing. The mold, the top and bottom plates and the screw clamps were conditioned at 23°C±1°C before testing.

The entire assembly was then transferred to an incubator for 1 hand maintained at 37°C±1°C. Then, the plates were removed and the surface of the specimen plane ends was prepared at right angles to the long axis, using an abrasive-coated paper and water. The specimen was then removed from the mold immediately after finishing and examined for air voids or chipped edges. Any specimen with these defects was discarded. Each acceptable specimen was then immersed in distilled water and maintained at 37°C±1°C in an oven for 24 h before testing.

About 24 h after completion the mixing, the compressive strength of the test specimens was determined using a Universal testing machine (Gunt, Germany) at a compressive strength mode equipped with a maximum load cell of 20 KN and running at a crosshead speed of 0.5 mm min^{-1}. The specimen was placed with the flat ends between the plates of the apparatus, so that, the load was applied along the long axis of the specimen. The load was applied using a custom-made cylindrical rod with a diameter corresponding to that of the specimen (Fig. 1).

Statistical analysis: Data are presented as the mean and standard deviation. The Student's t-test and Analysis of Variance (ANOVA) were used to investigate statistical differences, for all of these tests less than 0.05 was considered significant.

RESULTS

The mean setting time for the different experimental liquid formulae in the powder-to-liquid ratio is presented in Table 1.

Table 1: Setting time results of the different experimental formulae

Liquid formula	No. of samples	Setting time (sec) 8 drops	10 drops
1CH[a]:8PP[b]/1SPOON	5	5.45*	8.8[c]
1CH:10PP/1SPOON	5	7.62	7.51
1CH:20PP/1SPOON	5	7.09	9.39

[a]Chitosan; [b]Polyphosphonic; [c]Second

Table 2: Mean and standard deviation of compressive strength in MPa, for the experimental material formulae

Sample No.	Liquid formula	Compressive strength (MPa) Mean±SD[a]
5	1spoon /1CH[b]:8PP[c] (8 drops)	25.60±0.515*
5	1spoon /1CH:8PP (10 drops)	22.22±1.236
5	1spoon /1CH:10PP (8 drops)	17.12±0.311
5	1spoon /1CH:10PP (10 drops)	16.02±0.228
5	1spoon /1CH:20PP (8 drops)	12.52±0.460
5	1spoon /1CH:20PP (10 drops)	10.38±0.589

[a]Standard deviation; [b]Chitosan; [c]Polyphosphonic acid

Table 3: ANOVA test of compressive strength of the experimental material, glass ionomer cement and biodentin groups

	ANOVA				
Groups	Sum of squares	Df	Mean square	F-value	Sig.
Between groups	280.662	2	140.331	4.631	0.032
Within groups	363.652	12	30.304		
Total	644.314	14			

The setting time of the powder-to-liquid ratio for 1spoon/1CH:8PP of 8 drops was the best formula compared with the other formulae.

For compressive strength, the mean values and standard deviation recorded for different groups are presented in Table 2.

Table 2 shows that the compressive strength of the formula that contained 1 spoon of experimental powder with 8 drops of the solution and 1drop chitosan with 8 drops polyphosphonioc acid was 25.60±0.515 MPa, which was higher than that of the other formulae.

Statistical analysis using ANOVA revealed that there was a significant difference ($p \leq 0.05$) in the compressive strength between the formula containing 1 spoon/1 CH:8PP with8 drops compared with the biodent in group but there was no significant difference compared with the glass ionomer cement group (Table 3).

DISCUSSION

Setting time characteristics were associated with the reaction rates and they affect the practical application of many materials in restorative dentistry. Cement material depends on a critical reaction time and hardening rate for their successful application. For successful manipulation and application, the time required for a material to set or harden from a plastic or liquid state may be the most important quality.

The setting time for the experimental cement-based capping material was 5.45 min which is acceptable according to the ANSI/ADA Specification No. 30 for temporary restoratives and bases[11]. This setting time indicates that the experimental cement can be used as a base material.

Strength properties of a material define as its ability to resist disintegration during function. Adequate compressive strength ensures integrity of the adhesive joint under a vertical functional load. Strength properties of crystalline structures such asset cement are related to the crystal lattice structure and resistance offered by this structure against compressive forces of disintegration in the vertical plane[12].

Based on the results of the present study, a significant difference between the experimental cement group and the biodent in group was observed with no significant difference compared to the glass ionomer cement group.

The improvements in mechanical properties of the experimental cement-based capping material may indicate increased homogeneity and poly-salt bridge formation in the final set material[13].

Stronger bonds between the organic and inorganic networks most likely caused the increase in strength of the set cement. By increasing the degree of cross-linking through increasing the poly-salt bridge formation, the mechanical properties improve considerably. This, in turn, might make the material a better choice for posterior tooth restoration and as a bone grafting material in stress-bearing areas compared with the other materials[14].

This result suggests that the calcium ions reacted with the polyphosphonoic acid groups and these reactions were considered to form strong bonds with the polyacid salt matrix. The experimental cement-based capping material contained a low chitosan percentage which provides greater strength to the formula. This result is in agreement with Petri *et al*.[15] Biodentine was a stronger material tested compared with the experimental cement and glass ionomer cement, based on the compressive strength property. The increased strength is attributed to the low water-to-cement ratio used in biodentine which is permissible because a water-soluble polymer is added to the mixing liquid[16].

CONCLUSION

Within the limitations of the experimental methods used in the present study, the cement-based capping material prepared from egg shells is acceptable and within the limitations of ANSI/ADA Specification No. 30 for setting time and compressive strength values.

REFERENCES

01. Gandolfi, M.G., P. Taddei, F. Siboni, E. Modena, E.D.D. Stefano and C. Prati, 2011. Biomimetic remineralization of human dentin using promising innovative calcium-silicate hybrid smart materials. Dent. Mater., 27: 1055-1069.
02. Mallmann, A., J.C.O. Ataide, R. Amoedo, P.V. Rocha and L.B. Jacques, 2007. Compressive strength of glass ionomer cements using different specimen dimensions. Braz. Oral Restorative, 21: 204-208.
03. King'ori, A.M., 2011. A review of the uses of poultry eggshells and shell membranes. Int. J. Poult. Sci., 10: 908-912.
04. Murakami, F.S., P.O. Rodrigues, C.M.T.D. Campos and M.A.S. Silva, 2007. Physicochemical study of CaCO3 from egg shells. Food Sci. Technol., 27: 658-662.
05. Hennen, W.J., 1996. Chitosan. Woodland Publishing Inc, Salt Lake City, Utah, USA.,.
06. Kim, S.K., 2011. Chitin, Chitosan, Oligosaccharides and their Derivatives: Biological Activities and Applications. 1st Edn., CRC Press, Boca Raton, Florida, USA., ISBN-13: 978-1439816035, Pages: 666.
07. Engin, B., H. Demirtas and M. Eken, 2006. Temperature effects on egg shells investigated by XRD, IR and ESR techniques. Radiat. Phys. Chem., 75: 268-277.
08. Salman, R.F., 2006. Evaluation and assessment of new restorative filling material (polyphosphonate glass ionomer). Ph.D. Thesis, University of Baghdad, Baghdad, Iraq.
09. El-Hefian, E.A., E.S. Elgannoudi, A. Mainal and A.H. Yahaya, 2010. Characterization of chitosan in acetic acid: Rheological. Turk.. J. Chem., 34: 47-56.
10. Al-Khalidi, E.F., T.H. Al-Salman and A.A. Taqa, 2015. Preparation and evaluation of a new Calcium based cement. Ph.D. Thesis, University of Mosul, Mosul, Iraq.
11. American National Standard, 1990. Zinc oxide eugenol cements and Zinc oxide non eugenol cements. American Dental Association Specification No.30, American National Standards Institute, New York, USA.
12. Mandhyan, D., N. Mehrotra and Ravikant, 2012. A comparative evaluation of compressive strength, tensile strength and bond strength of various dental materials. J. Recent Adv. Applied Sci., 27: 57-61.
13. Wu, W., D. Xie, A. Puckett and J.W. Mays, 2003. Synthesis of amino acid-containing polyacids and their application in self-cured glass-ionomer cement. Eur. Polym. J., 39: 959-968.
14. Kao, E.C., B.M. Culbertson and D. Xie, 1996. Preparation of glass ionomer cement using N-acryloyl-substituted amino acid monomers-evaluation of physical properties. Dent. Mater., 12: 44-51.
15. Petri, D.F.S., J. Donega, A.M. Benassi and J.A.J.S. Bocangel, 2007. Preliminary study on chitosan modified glass ionomer restoratives. Dent. Mater., 23: 1004-1010.
16. Grech, L., B. Mallia and J. Camilleri, 2013. Characterization of set intermediate restorative material, Biodentine, bioaggregate and a prototype Calcium silicate cement for use as root-end filling materials. Int. Endodontic J., 46: 632-641.