OPEN ACCESS JOURNAL

BIOINTERFACE RESEARCH IN APPLIED CHEMISTRY

ORIGINAL ARTICLE

www.BiointerfaceResearch.com

ISSN 2069-5837

Volume 1, Issue 3, 2011, 083-088

Received: 21.04.2011 / Accepted: 28.05.2011 / Published on-line: 15.06.2011

Particle size of two endodontic biomaterials and Portland cement

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ABSTRACT

We aimed to analyze particle size of a new endodontic biomaterial [calcium enriched mixture (CEM) cement], white mineral trioxide aggregate (WMTA), and white Portland cement (WPC). The analyses were performed twice. For each analysis, 0.05 mg of test material was experimented using particle size analyzer model HELOS and disperser CUVETTE. Distribution of particles in different ranges in addition to cumulative percentage and the mean of particle size were calculated. Data were analyzed using one-way ANOVA, Tukey and Chi-square tests. No significant differences were observed between the cumulative percentages of particle size in test materials. However, means of particle size were significantly different between WPC and WMTA ($P<0.001$). Among different investigated ranges of particle size distribution, the range of ≤30µm showed significant difference between three tested materials ($P<0.05$). The largest distribution of smallest range of particles was related to CEM cement. The sealing ability and satisfying physical properties of this novel biomaterial are due to a high percentage of small partcles in CEM cement.

Keywords: *Calcium enriched mixture, CEM cement, endodontics, mineral trioxide aggregate, particle size, Portland cement*

1. Introduction

Particle size can influence different characteristics of biomaterials. Smaller sizes of particles have been shown to increase the exposed surface area leading to greater dissolution during setting reaction [1]. The increased exposed area is shown to facilitate the decrease in working/setting time. These were found in a study which used particle size analysis (PSA) to determine particle size and X-ray photoelectron spectroscopy (XPS) for investigating the structure and composition of the glass ionomer cement [2]. Particle size distribution has been introduced as a major factor to improve mechanical properties [3].

The early work of Kent and Wilson [4] has been continued by Brune and Smith [5] who investigated particle size based on sieve techniques; particle size has little effect on compressive strength. The increased abrasion resistance is associated with a decrease in particle size [6]. Having larger mean particle size has been also recognized as a contributing factor to the relative weakness of the material [3-7]. Finally a study showed that those materials composed of large particles (~10µm) formed a clay-like, non-cohesive paste, while those composed of finer particles (3.4µm) were strong but too

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fast-setting and viscous for clinical usage [1]. It has been also shown that smaller particles of materials appear to result in higher Compressive Strength (CS) and Diametral Tension Strength (DTS) [8]. All these and other different mechanical properties have been previously investigated [9-10]. Laser diffraction/image analysis is one of particle size analysis methods [11]. Dry/wet dispersion are two modules used in this analysis. Dry dispersion is used for dry powders while wet dispersion is suitable for suspensions and emulsions [12]. For extremely small quantities of valuable products and if pumping might destroy the particles or droplets, the dispersing module CUVETTE is best suited. Two versions, 50mL and 6mL cover the particle size range from 0.1-3500μm [12].
Particle size of materials has been also appraised via SEM [8,13]. It has been reported that with a similar particle size, a higher mechanical strength is designed by a reduced spreading in grid size [8,14]. Mineral trioxide aggregate (MTA) is a root-end filling material introduced in 1993 [15-16]. Currently, there are four available types of MTA including ProRoot MTA and MTA Angelus in gray and white forms. MTA is mainly composed of Portland cement (PC) [17]. ProRoot MTA has a particle distribution similar to PC [18]. On the other hand, it has been demonstrated that PC includes a wide range of particle sizes, whereas ProRoot MTA shows homogeneous image with equal particle size [13]. There are some clinical disadvantages for MTA such as extended setting time, poor handling and high price [19-21]. The manufacturer recommends mixing MTA with sterile water. This produces a granular, sand-like mixture that is difficult to be delivered to the required site and hard to condense adequately [21]. Several studies have shown that MTA and PC are very similar in chemical/physical character [22-24]. Studies of MTA and PC have found that these materials were biocompatible [25-26]. The handling characteristic of PC depends on its particle size and shape [27]. Other studies showed that handling characteristics of ceramics and polymers is improved by particle modification [28-29]. Calcium Enriched Mixture (CEM) consisting of different calcium compounds was recently developed by the first author. It has been shown that this biomaterial has good handling characteristics and shorter setting time than MTA. In addition, significantly superior results have been observed in film thickness and flow of CEM compared to MTA [30].
The aim of this *in vitro* study was to analyze the particle size of three different materials including White ProRoot MTA (WMTA), CEM, and White Portland cement (WPC).

2. Experimental section

Three types of materials including White Portland cement (WPC; Saveh Co. Saveh, Iran), White ProRoot MTA (WMTA; Lot 06002895, Dentsply, Tulsadental, USA), and CEM cement (BioniqueDent, Tehran, Iran) were used in this study. These samples were coded A-C and their particles' size were analyzed. For this purpose, particle size analyzer model HELOS and disperser CUVETTE with range of measurement between 0.1-3500 μm were used. This analyzer is technically used for emulsions and suspensions via wet technique in the range of 0.1-3500 μm. CUVETTE includes two 6-mL glass tubes (model SM) for particle size measurements of particles ranged between 0.1-35 μm (with R1 lens) and 50-mL (model US) for particle sizes ranged between 0.25-3500 μm (lens R2-R7). It also has a mixer for preventing sedimentation and ultrasound for dispersing particle. First of all, sample information and all measurement parameters such as reference time, measurement time, time and power of ultrasonic and also the mixture speed were recorded and saved. 50mL of alcohol (ethanol 90%) was pursed in glass tube model US. 0.05 gram of each sample was mixed with adequate alcohol, leading to a creamy mixture. This mixture was gradually added to the glass tube so that it reached the optical concentration between 15-27%. Then the measurements of particle size and dispersion were performed. The results were being presented as diagrams and tables. For more assurance, the measurements were performed twice and the two diagrams related to each sample were compared to each other. Three analytic tests were used in this study. The mean of particle size was measured for test materials using one-way ANOVA analysis. Tukey HSD test was used for pair comparison. In order to compare the distribution of particles in different ranges in different materials we used Chi-square test.

3. Results section

Cumulative percentage related to particle size of studied materials in this study showed no significant difference between WMTA, CEM cement, and WPC (Figure1). However, significant difference was observed between the mean particle sizes of test materials ($P<0.001$). Table 1 shows a descriptive statistical definition in addition to the means and standard deviations related to test materials. There is significant difference between the particle size of WMTA and WPC ($P<0.001$), while other pair comparisons did not show any significant difference.

Distribution of particles in the ranges of <10 µm, 10-20 µm, and 20-30 µm was not significantly different. However, the results related to distribution of particles≤30 µm and >30 µm showed significant difference between three tested materials ($P<0.05$).

Table 1: Descriptive statistical definition (means, SD, and CI) of particle size of tested materials

	Number	Mean	Standard Deviation	95% Confidence Interval for Mean		Minimum	Maximum
				Lower Bound	Upper Bound		
WMTA	100	10.90	10.04	8.91	12.89	0.60	51.0
WPC	100	19.21	20.28	15.19	23.24	0.60	103.0
CEM	100	14.19	14.98	10.99	17.19	0.60	88.0

Table 2: Distribution of particle sizes between 0.5-30 µm related to tested materials

Size range (µm)	WMTA		WPC		CEM cement	
	Count	Percent	Count	Percent	Count	Percent
0.5-2.5	17	17.2	15	15.2	25	19.0
2.6-4	12	12.1	9	9.1	13	11.4
4.1-6	15	15.2	10	10.1	11	11.5
6.1-15	31	31.3	24	24.2	18	24.9
15.1-30	19	19.2	21	21.2	21	20.3

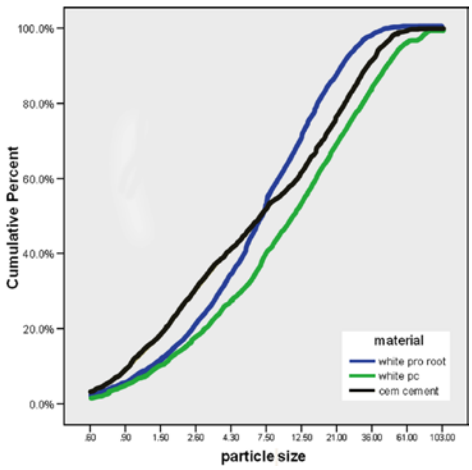

Figure 1: Cumulative percentage related to particle size of tested materials

Table 2 demonstrates the distribution of particle sizes between 0.5-30 μm related to each tested material. Data shows that the biggest distribution of particles with the range of 0.5-4 is related to CEM cement (Figure 2). All materials tested in this in vitro study were water based; in this regard, mixing these materials with water would induce hydration reactions. Therefore, alcohol was used for making a suspension through which particle detection as well as particle size measurements was possible. This type of suspension is in agreement with other particle size studies [18-19]. The density and size of dentin tubules in tooth root has been previously investigated [31]. It has been reported that the density and direction of dentin tubules at the apical portion of human teeth were irregular [32]. Generally, the average of considered diameter for dentin tubules is of 2-5 μm. Dentin is a substrate and dental cements are materials; therefore, the size of dentin tubules is in correlation with particle size of materials. Materials with particles in smaller sizes than dentin tubules are able to penetrate through these tubules and this can be an important mechanism for providing a hydraulic seal via a three dimensional seal [19,33]. The penetrating particles with high alkalinity through dentin tubules may act as the source of ion release, resulting in a high local pH with a slight chance of being reduced by dentin buffering [34]. Higher pH would result in more effective antibacterial activity. Studies on CEM cement demonstrated that this material is capable of phosphorus and calcium ions release, this biomaterial and also MTA contains calcium hydroxide [35]. All these can be in favor of a high antimicrobial activity of CEM cement [36].

Our study showed significant difference between the particle size of Portland cement and ProRoot MTA; this is in agreement with preceding investigation on comparative particle size measurement of these two materials [18]. Out study shows that the most distributed particle size in CEM cement was in the range of 0.5-2.5 μm (25.7%). This can lead to penetration of its particles in dentin tubules. It was also showed that at the range of 0.5-2.5 μm, the biggest distribution of particles was related to CEM cement, like a previous report [37].

This can be supported by a previous study which showed better, but not significant, sealing ability of CEM cement than that of MTAs [39]. High presence of small size particles in CEM cement may also explain the shorter setting time, better flow and also less film thickness of this dental material which has been demonstrated previously [30].

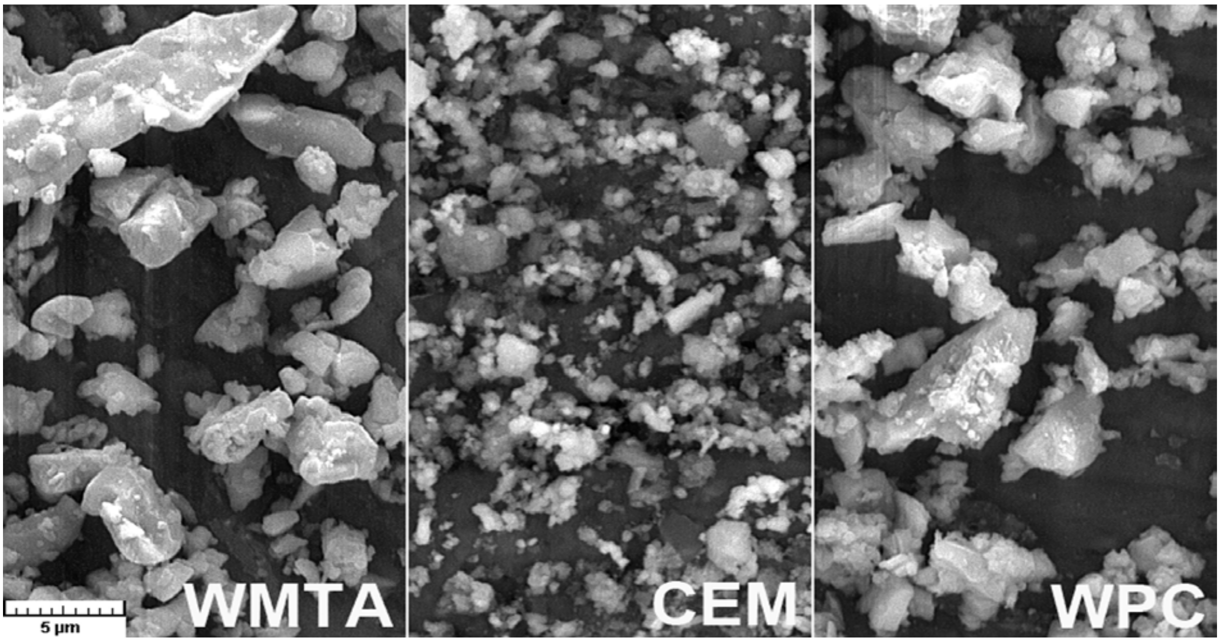

Figure 2: Scanning electron photomicrograph of tested materials: A) white mineral trioxide aggregate, B) calcium enriched mixture (CEM) cement, and C) white Portland cement (Mag ×5000, Bar = 5μm).

4. Conclusions

Small-size particles in CEM cement were significantly more than in two other materials including WMTA and WPC which were similar to each other. In this regard, superior sealing ability and high physical properties of CEM cement can be explained. Therefore, this new endodontic biomaterial can be an acceptable alternative for MTA in clinical usage; however, further investigations are required for more clarification of other properties of this material.

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Acknowledgement: This study was supported by Iranian Center for Endodontic Research, Shahid Beheshti Medical University, Tehran, Iran. We thank Mr. Majid Madani for his assistance in particle size analysis, and also Dr. Alireza Akbarzadeh for statistical analysis