NATIONAL BUREAU OF STANDARDS REPORT

9804

Progress Report

on

ALUMINUM OXIDE AS A REINFORCING AGENT FOR ZINC OXIDE–EUGENOL–EBA CEMENTS

NBS

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

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Aluminum Oxide as a Reinforcing Agent for Zinc Oxide-Eugenol-EBA Cements

G. M. Brauer, R. P. McLaughlin and E. F. Huget

Aluminum oxide is a very effective reinforcing agent for o-ethoxybenzoic acid (EBA) cements. Addition of Al_2O_3 increases the amount of powder that can be incorporated into the mix. The compressive strength of the hardened cement is increased up to 1055 kg/cm^2 (15,000 psi) and the ADA film thickness decreased to 26μ. The materials adhere to tooth structure as well as zinc phosphate cements and are suitable as crown and bridge cements. With higher powder-liquid ratios their high ten-minute compressive strength and excellent tissue tolerance suggests their use as bases under metallic restorations. These materials may also be employed as temporary restoratives. Mixes of Al_2O_3 and eugenol or glycerine may be of interest as a temporary non-hardening crown and bridge cement.

Incorporation of Al_2O_3 whiskers did not improve the physical properties of these cements.

1. INTRODUCTION

The partial replacement of eugenol by o-ethoxybenzoic acid (EBA) in zinc oxide-eugenol (ZOE) cements has been shown to yield greatly improved products[1-3]. Results of these investigations have led to the development of a biologically acceptable crown and bridge cement consisting of a powder composed of ZnO, hydrogenated rosin, fused quartz, and EBA-eugenol liquid. A number of commercial products employing these formulations having optimum physical properties have recently become available. Alumina is often used as a reinforcing agent in ceramics, Since it is considerably more rigid than fuxed quartz[4,5], the present study was undertaken to determine if alumina reinforcement would improve the properties of EBA dental cements.

2. EXPERIMENTAL

2.1. Materials*

Zinc oxide, reagent grade, was passed through a No. 80 sieve. Crushed hydrogenated rosin** was passed through a No. 100 sieve. Unless otherwise specified, "tabular alumina"*** was used. The particle size range of this alumina varied from $<1\mu$ to $>20\mu$, with very few particles $>20\mu$. Smallest particles were barely visible under the microscope ($<0.5\mu$). Particles were irregular in shape and most grains were thinner in one direction than in the other two. This material was

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* Certain commercial materials and equipment are identified in this paper in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, ncr does it imply that the material or equipment is necesarily the best available for the purpose.

** Staybelite, Hercules, Inc., Wilmington, Delaware

*** T61 Tabular Alumina, Aluminum Co. of America, Bauxite, Ark.

heated to 700°C for two hours, passed through a No. 400 sieve and cooled. An irregular shaped "calcined alumina"* was also used in two formulations. It contained particles ranging from 24μ to those barely visible under the microscope (<0.5μ). This alumina was also heated to 700°C. Whiskers consisting of loose sapphire (Al_2O_3) fibers of varying particle size**, sapphire submicron blades**, silicon carbide fiber crystals**, aluminum nitride-oxide fiber crystals and silicon carbide fibers*** were incorporated into the powder. The dimensions of the whiskers are given in Table 6. Poly(methyl methacrylate) powder, stearic acid (USP) and talcum (USP) were passed through a No. 70 sieve, aluminum sulfate (reagent grade), zinc stearate (technical grade), a highly stabilized rosin**** and two highly stabilized ester rosins", were sieved through a No. 100 sieve.

EBA and eugenol were reagent grade, glycerine USP grade, and the distilled tall oil technical grade.

The powders were mixed by tumbling weighed amounts of the constituents in glass jars. Unless stated otherwise, the liquid employed contained 62.5 percent EBA and 37.5 percent eugenol.

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* A-2 Calcined Alumina, Aluminum Co. of America, Bauxite, Ark.

** Thermokinetic Fibers, Inc., 136 Washington Ave., Nutley, N.J.

*** The Carborundum Co., Niagara Falls, New York.

**** Floral AX, Hercules, Inc., Wilmington, Delaware.

" Floral 85 and Floral 105, Hercules, Inc., Wilmington, Del.

2.2 Methods

The powder and liquid were mixed on a glass slab. A mortar and pestle were used for those mixes that could be mixed only with difficulty by spatulation. One formulation was also mixed for 30 seconds in a capsule containing a 9/32 inch steel ball in a mechanical amalgamator. Consistency, setting time, film thickness, one-week compressive strength, and solubility and disintegration were determined according to American Dental Association Specification No. 8[6]. A Tinius-Olsen pendulum type testing machine was used to determine compressive strength. Consistency and one-day solubility and disintegration values were obtained employing American Dental Association Specification No. 9 (dental silicate cements)[7] for materials that were considered suitable for possible application as bases or restorative materials. The technique of Oldham, Swartz and Phillips[8] was employed in the study of tensile adhesion of these cements. Two series of five teeth each were prepared to receive one-surface inlays, and an average value for tensile adhesion was obtained from the five runs. In series 2, the order in which the adhesion values of the various materials were measured was the reverse of that used in series 1. This arrangement was employed to compensate for any decrease in adhesion because of changes in the dentinal surfaces on repetitive testing.

To evaluate the aluminum oxide-reinforced EBA cements as a base, 1.7 Gm of powder containing 30 percent tabular Al_2O_3, 6 percent hydrogenated rosin, and 64 percent zinc oxide were mixed with 0.2 ml of liquid and placed as a base under a series of amalgam restorations which were placed with a packing pressure of 140 kg/cm^2 (2,000 lb/in^2) using a calibrated spring plugger. The teeth were sectioned after 48 hours to determine if the bases were capable of withstanding this packing pressure.

3. RESULTS

For comparison, the physical properties of commercial zinc oxide-eugenol (ZOE), EBA cements reinforced with 20 percent fused quartz, and zinc phosphate cements are given in Table 1. The composition of the commercial EBA cement and its physical properties were nearly the same as those reported earlier for an experimental cement.[1] Increasing the powder-liquid ratio of the EBA cement from 1.4 to 1.5 Gm/0.2 ml increased the compressive strength from 740 to 810 kg/cm^2. The solubility and disintegration values of the EBA cement were considerably lower than those of the ZOE and zinc phosphate cements. On substitution of Al_2O_3 for fused quartz (Table 2), more powder could be incorporated into the mix, mixing properties and compressive strength were improved and film thickness was greatly reduced. Better mixing characteristics and somewhat higher compressive strengths were obtained with the tabular than with the calcined alumina. When the percentage of Al_2O_3 was varied between 20 and 40 percent, the physical properties reached a maximum with a cement containing 30 percent tabular Al_2O_3. Using a powder-liquid ratio of 1.7 Gm/0.2 ml, an easily mixed slurry was obtained which on hardening yielded a product with a one-week compressive strength of 955 kg/cm^2 (13,600 psi), a solubility and disintegration value of 0.05 percent, and a film thickness of 26μ. The EBA cements could also be mixed efficiently in a mechanical amalgamator. The resulting mixes had a lower consistency, but otherwise physical properties very similar to those obtained on hand spatulation using identical powder-liquid ratios. As much as 2.1 Gm powder per 0.2 ml liquid was easily incorporated by mechanical mixing. This product, exhibited the best physical properties obtained for any EBA cement, having a one-week compressive strength of 1055 kg/cm^2 (15,000 psi) and a solubility and disintegration of 0.03 percent. With a 1.7 Gm/0.2 ml powder-liquid ratio, a 10-minute compressive strength of 470 kg/cm^2 (6660 psi) was obtained. A mix utilizing a USP zinc oxide that had been stored for over ten years set very fast (4.5 minutes) and had a low consistency value. Analysis indicated that this particular ZnO contained a considerable amount of zinc carbonate. Since cements incorporating this zinc oxide showed promise as possible restorative materials, small amounts of sodium bicarbonate and water were added to freshly procured USP zinc oxides. However, the resulting mixes had too low a consistency value for clinical application.

The effect of addition of rosin derivates on tabular Al_2O_3-reinforced EBA cements is shown in Table 3. Cements containing stabilized rosin, rosin esters, or abietic acid (the major constituent of rosin) had good mixing characteristics and low film thickness. However, the addition of the rosin derivatives increased the solubility and decreased the compressive strength of the resulting products. The addition of hydrogenated rosin up to eight percent enhanced the mixing characteristics, reduced solubility and disintegration, but increased setting time from 5 to 10 minutes. The compressive strength decreased when the hydrogenated rosin content was greater than 2 percent.

Other additives modified the properties of the reinforced EBA cements (Table 4 and 5). Stearic acid, zinc stearate, and talcum decreased the consistency values and hence are useful in the formulation of base or restorative materials where high film thickness is of no importance. Stearic acid and tall oil improved the mixing properties, but lowered compressive strength. Addition of 0.5 to 1 percent zinc stearate slightly increased the solubility and disintegration. The addition of 0.5 to 1 percent aluminum sulfate slightly lowered the compressive strength and the solubility and disintegration. Incorporation of 4 percent methyl methacrylate polymer powder did not prove beneficial.

Partial replacement of the tabular Al_2O_3 particles by

Al_2O_3 whiskers or silicon carbide whiskers did not result in any improvement of the physical properties (Table 6). Mixing characteristics were generally poor, but could be improved by the addition of 0.5 percent stearic acid Cements with sapphire (Al_2O_3) gave higher compressive strengths than those containing silicon carbide whiskers. With sapphire (Al_2O_3) a slight increase in compressive strength was obtained with decreasing particle diameter.

The tensile adhesion measurements gave larger standard deviations than those experienced on measuring compressive strength values (Table 7). The EBA cements adhered at least as well as commercial zinc phosphate and much better than ZOE cements. There was no significant difference between the tensile adhesion of tabular aluminum oxide and fused quartz reinforced EBA cements. Rupture of the specimens always occurred at the cement-gold interface with the Al_2O_3-reinforced cements rather than at the cement-dentin interface where all the other materials failed in tension. A larger powder-liquid ratio of EBA cement produced slightly improved adhesion. Mixes of zinc oxide or aluminum oxide-reinforced powders with water did not produce any significant adhesion between inlay and tooth surface. Thus, the liquid reactant in the respective cements was necessary to produce adhesion.

When thin slurries of aluminum oxide were mixed with either eugenol or glycerine and placed between glass plates, these plates could only be separated with difficulty even after immersion in water for several months. Thus, such mixes may prove useful as non-hardening temporary crown and bridge cements.

Figure 1 shows an MOD amalgam restoration placed over an aluminum oxide-reinforced EBA cement. The base is still intact whereas ZOE bases fractured at the pulpal-proximal line angle.

4. DISCUSSION

Aluminum oxide-reinforced EBA cements have physical properties superior to those which are reinforced with fused quartz. The preferred cement contained 30 percent tabular Al_2O_3 and 6 percent hydrogenated rosin. Using 1.7 Gm powder per 0.2 ml liquid, a slurry can be mixed easily and hardens in less than ten minutes. The resulting product has a compressive strength of 950 kg/cm^2 and film thickness of 26μ. These properties make the product very desirable for use as crown and bridge cements. On incorporation of more powder into the mix, excellent base materials can be obtained. The products have physical properties much superior to those of conventional ZOE cements. Especially desirable is their high 10-minute compressive strength which can easily withstand the forces encountered in condensing an amalgam.

Most additives investigated did not improve the properties of the cements. When incorporated in EBA cements, aluminum sulfate decreased the water solubility and disintegration of the cements within the limits of the experimental error associated with this test. Addition of poly(methyl methacrylate) did not improve the properties of the resulting cement. Materials containing a polymer should be more resilient. This would be advantageous in formulating a useful restorative material. Thus, further studies should be made to determine possible beneficial effects of polymeric fillers.

Alumina whiskers have a tensile strength that is a whole order of magnitude greater than aluminum oxide in bulk form, but whisker reinforcement is dependent on a number of parameters including proper alignment and uniform distribution of fibers and their complete wetting and bonding to the matrix. If these conditions are met by the composite, the load or stress can be transferred through the "weak" matrix to the "strong" fibers which have a much higher elastic modulus. The surface area of whiskers is very large. Using a fixed (minimum) amount of EBA-eugenol liquid, the whisker concentration in the powder must be kept low to retain good mixing characteristics and to obtain complete bonding between matrix and fiber. Since whiskers were not found to improve the finished composites, their concentration in the cement may have been insufficient or any of the prerequisites discussed above such as complete wetting of the surface may not have been accomplished. Clustering of the fibers on dry mixing of whiskers with the remaining powder also caused difficulties. This clustering is the result of electrostatic surface interaction and might be overcome by proper pretreatment.

Mixes having a low consistency value may be useful as a temporary restorative material. Clinical studies to determine the effective service life of these compositions are in progress.

Of interest would also be the application of a temporary non-hardening crown and bridge cement consisting of a mixture of Al_2O_3 and eugenol or glycerine. The advantages of such a cement would be to facilitate (1) periodic observation of clinical crowns of abutment teeth, (2) periodic vitality testing of abutment teeth requiring full coverage, (3) post-insertion root canal therapy without the involvement of the restoration, (4) refabrication of pontics to obtain better ridge relationships for immediate insertion cases, (5) realignment of components to "improve" esthetics or function, and (6) equilibration and polishing.

5. CONCLUSIONS

Cements reinforced with tabular Al_2O_3 yielded higher compressive strengths and lower film thicknesses than those containing fused quartz. Physical properties including tensile adhesion of Al_2O_3 reinforced cements were in the same range as those of zinc phosphate cements. Aluminum oxide reinforced EBA cements should be very desirable for the cementation of crowns and bridges, and for bases under metallic fillings. They may also find application as temporary restoratives. Mixes of Al_2O_3 and eugenol or glycerine may be useful as temporary non-hardening crown and bridge cements.

The addition of untreated commercial Al_2O_3 whiskers did not improve the properties of the hardened cements.

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Fig. 1. Section through an amalgam restoration condensed under 140 kg/cm^2 (2000 psi) packing pressure against an Al_2O_3 reinforced base having a 10-minute compressive strength of 470 kg/cm^2 (6800 psi).

TABLE 1

Physical Properties of Commercial Cements

	ZOE	EBA* Pow.-Liq. Ratio gm/0.2 ml 1.4	1.5	Zinc Phosphate
Mixing Properties	good	good	fair	good
Consistency, mm.	---	41	39	30
Setting Time, Min.	7-8	8.5	8.5	7-8
One-Day Compressive Strength, kg/cm^2	140-385	670	750	1050
One-Week Compressive Strength, kg/cm^2	260	740	810	840-1220
Solubility and Disintegration, %	0.10	0.04	---	0.10-0.30
Film Thickness, μ	---	40	39	<40

*Opotow - EBA Crown and Bridge Cement, Buffalo Dental Manufacturing Co., Brooklyn, New York

TABLE 2

Properties of Al_2O_3-Reinforced EBA Cements

Liquid Composition: 62.5% EBA - 37.5% Eugenol

Powder Composition			Powder-Liquid Ratio	Mixing Properties	Consistency	Setting Time	Compressive Strength		Solubility and Disintegration	Film Thickness
ZnO	Al_2O_3	Hyd. Rosin					One Day	One Week		
%	%	%	Gm/0.2ml		mm.	min.	kg/cm²	kg/cm²	%	μ
74	20	6	1.7	good	48	9.5	800	870	0.04	25
74	20	6	2.1	fair	40(44)*	9.5	880	990	.04	28
74	20	6	2.6	very poor	30	--	830	---	---	--
74	20 †	6	2.1	fair	40(44)*	9	820	---	---	26
74	20 †	6	2.6	poor	30(36)*	8.5	840	---	---	--
69	25	6	1.7	good	--	9.5	810	---	---	--
64	30	6	1.7	good	47	9.5	870	955	.05	26
64	30	6	1.7	‡	26	9	860	940	---	35
64	30	6	2.1	‡	19(25)*	8.5	930	1055	.03	48
64§	30	6	1.5	fair	22*	4.5	920	---	.05	--
59	35	6	1.7	good	--	9	850	---	---	--
54	40	6	1.7	good	--	8.5	780	---	---	--

*Consistency determined by ADA Specification No. 9
†Particle size <24μ
‡Mixed in a mechanical amalgamator for 30 seconds
§ZnO (USP) containing carbonate

TABLE 3

Effect of Rosin Content on Physical Properties of

Al_2O_3-Reinforced EBA Cements

Liquid Composition: 62.5% EBA - 37.5% Eugenol; Powder-Liquid Ratio: 1.7 Gm/0.2 ml

Powder Composition ZnO	Al$_2$O$_3$	Hyd. Rosin	Mixing Proper-ties	Consis-tency	Setting Time	Compressive Strength One Day	One Week	Solubility and Disintegration	Film Thick-ness
%	%	%		mm.	min.	kg/cm^2	kg/cm^2	%	μ
74	20	6	good	48	9.5	800	870	0.04	25
74	20	6*	good	--	8.5	780	---	35.6	--
74	20	6†	good	--	---	795	---	---	--
74	20	6‡	fair	45	9	790	850	0.19	26
74	20	6§	fair	--	---	780	---	---	--
70	30	0	poor	--	5	820	---	---	--
68	30	2	poor	42	7.5	930	---	0.30	38
66	30	4	good	45	9.5	900	---	0.08	26
64	30	6	good	47	9.5	870	955	0.05	26
62	30	8	very good	--	10	775	---	---	--

*Abietic acid
†Stabilized rosin (Foral AX)
‡Ester Resin (Foral 105)
§Ester Resin (Foral 85)

TABLE 4

Effect of Additives on Physical Properties of

Al_2O_3-Reinforced EBA Cements

Composition of Liquid: 62.5% EBA - 37.5% Eugenol; Powder-Liquid Ratio: 1.7 Gm/0.2 ml

Powder Composition ZnO	Powder Composition Al_2O_3	Powder Composition Hyd. Rosin	Additive	Mixing Proper-ties	Consis-tency	Setting Time	Compressive Strength One Day	Compressive Strength One Week	Solubility and Disin-tegration	Film Thick-ness
%	%	%			mm.	min.	kg/cm²	kg/cm²	%	μ
74	19	6	1% Stearic acid	very good	28	8.5	770	830	0.09	47
74	19	6	1% Talcum	poor	32	9.5	800	---	---	43
74	16	6	4% Methyl Metha-crylate	fair	48	9.5	750	810	---	--
74	16	6	4% Methyl Metha-crylate*	poor	40	---	820	770	---	--
65	30	4	1% $Al_2(SO_4)_3$	fair	41	8.5	850	---	.05	30
65.5	30	4	0.5% $Al_2(SO_4)_3$	good	43	9	860	---	.05	29
65	30	4	1% Zinc Stearate	good	20†	8	830	---	.11	20
65.5	30	4	0.5% Zinc Stearate	good	27†	9	840	---	.09	27

*Powder-Liquid ratio: 2.1 Gm/0.2 ml

†Consistency determined by ADA Specification No. 9

TABLE 5

Effect of Addition of Tall Oil on Physical Properties of Al_2O_3-

Reinforced EBA Cements

Powder Composition: 74% ZnO, 6% Hydrogenated Rosin, 20% Al_2O_3

Liquid Composition			Powder-Liquid Ratio	Mixing Properties	Consistency	Setting Time	1-Day Comp. Strength	Film Thickness
EBA	Eugenol	Tall Oil						
%	%	%	Gm/0.2 ml		mm.	min.	kg/cm^2	μ
56.3	33.7	10	2.1	very good	29	9	670	49
56.3	33.7	10	2.9	poor	---	---	710	---
59.3	35.7	5	2.5	poor	---	---	660	---
62.5	34.5	3	2.1	poor	---	---	730	---
62.5	36.5	1	1.8	good	45	7.5	760	---

TABLE 6

Properties of EBA Cements Containing Whiskers

Composition of Liquid: 62.5% EBA, 37.5% Eugenol

Powder Composition					Powder-Liquid Ratio	Mixing Properties	Consistency	One-Day Compressive Strength
ZnO	Al_2O_3 20μ	Hyd. Rosin	Al_2O_3 Whiskers	Stearic acid				
%	%	%	%	%	Gm/0.2 ml		mm.	kg/cm^2
74	17.5	6	3.5*	---	2.1	Poor	40	880
74	17.5	6	3.5*	---	1.7	Good	49	---
74	16	6	4†	---	2.1	Poor	--	860#
74	16	6	4‡	---	2.1	Poor	--	850#
74	17	6	3§	---	1.7	Fair	50	810
74	16	5.5	4§	0.5	1.8	Good	32	790
74	16	5.5	4§	0.5	1.4	Good	--	780
74	16	6	4//	---	2.1	Poor	41	810
74	16	6	4¶	---	1.7	Fair	48	800
74	16	6	4	---	2.1	Poor	42	800

*Sapphire (Al_2O_3) diameter 0.2 - 1.0μ, length 2-20μ
†Sapphire (Al_2O_3), diameter 1-10μ, length 60-1250μ
‡Sapphire (Al_2O_3), diameter 1-30μ, length 180-2500μ
§Mixed AlN and Al_2O_3 ~~whiskers~~, diameter 3-30μ, length 30-600μ
//Silicon carbide, diameter 0.5-3μ, length 10-300μ
¶ Silicon carbide, diameter 1-5μ
#One-week compressive strength

TABLE 7

Tensile Adhesion

Material	Tensile Adhesion¶	
	Series 1 kg.	Series 2 kg.
ZOE*	2.4 ± 1.4#	---
EBA†	6.5 ± 1.5	6.6 ± 4.4#
Zinc Phosphate‡	7.0 ± 1.7	---
Zinc Phosphate §	6.4 ± 2.4	---
Zinc Phosphate″	5.3 ± 3.6	5.4 ± 2.7
EBA (74% ZnO, 20% Al_2O_3, 6% Hyd. Rosin)	---	---
Powder-Liquid ratio 1.2 gm/0.2 ml	5.4 ± 1.5	---
Powder-Liquid ratio 1.7 gm/0.2 ml	6.2 ± 2.0	6.6 ± 3.2
Powder (74% ZnO, 20% Al_2O_3, 6% Hyd. Rosin) + H_2O	---	0.1 ± 0.0
Zinc Oxide + H_2O	---	0.1 ± 0.2

*S. S. White ZOE Cement, S. S. White Co., Philadelphia, Pennsylvania
†Opotow EBA Crown and Bridge Cement, Buffalo Dental Manufacturing Co., Brooklyn, New York
‡Lang Crown Bridge and Inlay, Lang Dental Manufacturing Co., Chicago, Illinois
§ Modern Tenacin, The L. D. Caulk Co., Milford, Delaware
″S. S. White Zinc Cement Improved, S. S. White Co., Philadelphia, Pennsylvania
¶Average of 5 runs, cross head speed of Instron testing machine 0.01 in/min.
#Standard deviation

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