NATIONAL BUREAU OF STANDARDS REPORT

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Silicate Cement: Method of Mixing in a Closed Container to Prevent Effects of Exposure to Atmosphere

by

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U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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THE NATIONAL BUREAU OF STANDARDS

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2. **OPTICS AND METROLOGY.** Photometry and Colorimetry. Optical Instruments. Photographic Technology. Length. Gage.
3. **HEAT AND POWER.** Temperature Measurements. Thermodynamics. Cryogenics. Engines and Lubrication. Engine Fuels.
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10. **BUILDING TECHNOLOGY.** Structural Engineering. Fire Protection. Heating and Air Conditioning. Exterior and Interior Coverings. Codes and Specifications.
11. **APPLIED MATHEMATICS.** Numerical Analysis. Computation. Statistical Engineering. Machine Development.
12. **ELECTRONICS.** Engineering Electronics. Electron Tubes. Electronic Computers. Electronic Instrumentation.
13. **ORDNANCE DEVELOPMENT.** Mechanical Research and Development. Electromechanical Fuzes. Technical Services. Missile Fuzing Research. Missile Fuzing Development. Projectile Fuzes. Ordnance Components. Ordnance Tests. Ordnance Research.
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15. **MISSILE DEVELOPMENT.** Missile Engineering. Missile Dynamics. Missile Intelligence. Missile Instrumentation. Technical Services. Combustion.

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Silicate Cement: Method of Mixing in a Closed Container to Prevent Effects of Exposure to Atmosphere

Abstract

Since the properties of a silicate cement restoration are radically affected by the temperature at which it is mixed and by the water content of the cement liquid, it has been impossible to produce good restorations in a hot, humid atmosphere. To overcome these difficulties a method of mixing the cement in a closed, cooled container was developed. By the use of this method of mixing, satisfactory restorations can be produced under all climatic conditions.

1. INTRODUCTION

Silicate cement liquids, being essentially aqueous solutions of phosphoric acid, are hygroscopic. Consequently, when exposed to air the liquids take on or give off water, depending upon the amount of moisture present in the air. This gain or loss of water, which greatly affects the setting time of the cement, occurs principally when the cement liquid is exposed to air during withdrawal of liquid from the bottle, while the liquid rests on the glass slab prior to mixing, and during mixing of the powder and liquid. The cement liquid should therefore not be exposed to air if ideal conditions are to prevail in mixing silicate cements. Also, the temperature of the glass slab upon which the cement mix is prepared markedly affects the amount of powder that can be incorporated into a given quantity of liquid to produce a workable plastic mix. If the slab is cold, a high powder-liquid ratio can be obtained and the cement will be stronger and less soluble and will have less shrinkage than if a warm slab is used. Unfortunately, however, water will condense on the slab when it is chilled much below room temperature during hot and humid weather. It was the purpose of this investigation to devise a system which would eliminate exposure of the silicate cement liquid to the air during storage or mixing and would permit cement to be chilled and kept moisture free during mixing, independently of air temperature and humidity. If such a procedure were practicable, then restorations of silicate cement with high compressive strenth, low solubility and small shrinkage during setting could be made repeatedly, regardless of weather conditions.

2. METHODS OF MIXING

Several methods of mixing silicate cement in a sealed container, at the same time maintaining a high powder-liquid ratio, were investigated. The most satisfactory method developed consisted of mixing the cement in a sealed flexible container held under cold water. Thin-walled, uncolored, paddle-shaped rubber balloons were used as the mixing containers. A measured amount of powder ample for the large majority of restorations was inserted with the aid of a glass funnel into a washed and dried balloon. The balloon was cut off approximately one inch from the bottom, and the bottom portion containing the silicate powder was sealed with rubber cement.

It was found that better mixes were obtained if most of the air was extracted from the container after sealing in the powder. This was done routinely with a 24 gage needle and syringe. In order to eliminate any possible seepage of air into the balloon, a patch of balloon material was cemented over the point of insertion of the needle.

The liquid was easily protected from air by storing it in a small serum bottle with a self-sealing rubber stopper through which a needle could be repeatedly inserted and the liquid withdrawn as needed for injection into the powder container. The liquid was injected into the balloon through the double-wall thickness in the area of patch which provided a self seal. Mixing was begun immediately after injection of the liquid. The powder and liquid were kneaded rapidly between the fingers and thumb of both hands for one minute, under water.

Some attempts were made to simplify the method by sealing the liquid and powder in separate compartments of the mixing container in such a way that the liquid could be forced into the powder at the time of mixing, but it was found that the rubber balloons and rubber cement used for sealing were attacked by the silicate cement liquid at common storage temperatures. It is probable that a method of packaging eliminating the separate bottle of liquid could be developed by further work.

3. PROPERTIES OF CEMENT MIXED IN SEALED CONTAINERS

3.1 Powder-Liquid Ratio

The standard testing consistency of the cement used in this investigation, determined according to American Dental Association Specification No. 9, was 1.65 g of powder to 0.4 ml of liquid. The manufacturer's recommended consistency was 1.45 g per 0.4 ml.

At a normal room temperature of 21° C (70° F) and a relative humidity of 65 ± 10 percent mixes at a consistency of 1.7 g per 0.4 ml were made in sealed containers in water at 21° C with no difficulty, and a consistency of 1.9 g per 0.4 ml was possible. It was impossible to incorporate 1.9 g of powder in 0.4 ml of liquid on a slab even though it was cooled.

At a room temperature of 32° C (90° F) and a relative humidity of 90 percent it was impossible to incorporate 1.7 g of powder in 0.4 ml of liquid on a slab cooled to just above the dew point, 30.5° C (87° F). By mixing in the rubber container a ratio of 1.7 g to 0.4 ml was obtained in water at 20° C with no difficulty, even though the room temperature was 32° C (90° F).

3.2 Setting Time

In Table 1 are shown the times of setting of a silicate cement mixed in a sealed container in water at four different temperatures [15° C (59° F), 20° C (68° F), 25° C (77° F) and 30° C (86° F)] and tested at 21° C (70° F) and 32° C (90° F). Specimens used in these tests were mixed at a ratio of 1.7 g to 0.4 ml. The tests were conducted as specified in American Dental Association Specification No. 9, with the exceptions that mixing was done in the sealed container and that a mix one-half the size prescribed in the specification was used (0.85 g of powder to 0.2 ml of liquid).

As can be seen from Table 1, specimens mixed under water at temperatures ranging from 15° to 30° C (59° to 86° F) gave setting times of $3\frac{3}{4}$ to $2\frac{3}{4}$ minutes when the materials were stored and the specimens were prepared at a room temperature of 21° C (70° F) and a relative humidity of 65 ± 10 percent, and setting times of $2\frac{1}{2}$ to $1\frac{3}{4}$ minutes when stored and prepared at a temperature of 32° C (90° F) and a relative humidity of 90 ± 10 percent. All mixes made under water at 20° C (68° F) or less allowed ample time for proper insertion in the average restoration even though the atmospheric temperature and humidity were high.

Mixes were also made under water at 10° C (50° F) and 35° C (95° F). Water at 10° C (50° F) was found to be too cold for the hands. Mixes made under water at 35° C (95° F) set in the rubber bag during mixing.

Table 1

Setting Time of a Silicate Cement
Mixed Under Water in Sealed Containers

Temperature of Water		Setting Time*	
		21° C (70° F) Humidity 65±10%	32° C (90° F) Humidity 90±10%
°C	°F	minutes	minutes
15	59	$3\frac{3}{4}$	$2\frac{1}{2}$
20	68	$3\frac{1}{2}$	$2\frac{1}{2}$
25	77	$3\frac{1}{2}$	$2\frac{1}{4}$
30	86	$2\frac{3}{4}$	$1\frac{3}{4}$

*Setting time given is average of 3 mixes recorded to nearest 15 seconds from start of mix. Mix consisted of 0.85 g of powder to 0.2 ml of liquid.

4 CONCLUSIONS

The influence of atmospheric conditions upon the mixing of silicate cements can be eliminated to a large extent by mixing in a closed container. Thus a comparatively large amount of powder can be incorporated into a given quantity of liquid and satisfactory silicate cement restorations can be made under all climatic conditions.

THE NATIONAL BUREAU OF STANDARDS

Functions and Activities

The National Bureau of Standards is the principal agency of the Federal Government for fundamental and applied research in physics, mathematics, chemistry, and engineering. Its activities range from the determination of physical constants and properties of materials, the development and maintenance of the national standards of measurement in the physical sciences, and the development of methods and instruments of measurement, to the development of special devices for the military and civilian agencies of the Government. The work includes basic and applied research, development, engineering, instrumentation, testing, evaluation, calibration services, and various scientific and technical advisory services. A major portion of the NBS work is performed for other government agencies, particularly the Department of Defense and the Atomic Energy Commission. The functions of the National Bureau of Standards are set forth in the Act of Congress, March 3, 1901, as amended by Congress in Public Law 619, 1950. The scope of activities is suggested in the listing of divisions and sections on the inside of the front cover.

Reports and Publications

The results of the Bureau's work take the form of either actual equipment and devices or published papers and reports. Reports are issued to the sponsoring agency of a particular project or program. Published papers appear either in the Bureau's own series of publications or in the journals of professional and scientific societies. The Bureau itself publishes three monthly periodicals, available from the Government Printing Office: the Journal of Research, which presents complete papers reporting technical investigations; the Technical News Bulletin, which presents summary and preliminary reports on work in progress; and Basic Radio Propagation Predictions, which provides data for determining the best frequencies to use for radio communications throughout the world. There are also five series of nonperiodical publications: the Applied Mathematics Series, Circulars, Handbooks, Building Materials and Structures Reports, and Miscellaneous Publications.

Information on the Bureau's publications can be found in NBS Circular 460, Publications of the National Bureau of Standards ($1.00). Information on calibration services and fees can be found in NBS Circular 483, Testing by the National Bureau of Standards (25 cents). Both are available from the Government Printing Office. Inquiries regarding the Bureau's reports and publications should be addressed to the Office of Scientific Publications, National Bureau of Standards, Washington 25, D. C.

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