The Effect of Cold Temperature Mixing on the Properties of Zinc Phosphate Cement

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Although zinc phosphate cement has been successfully used in orthodontics for many years, it has two properties which may handicap its clinical use. First, the short working time of the cement sometimes does not permit more than a few orthodontic bands to be cemented at one time. Second, the setting time in the oral environment is too long to be clinically convenient; the orthodontist must often wait several minutes before excess cement can be removed. To minimize these undesirable properties many orthodontists are presently mixing zinc phosphate cement on a cooled or frozen glass mixing slab. Subjective opinions from these operators indicate that the cold slab mixing technique permits the cement to exhibit prolonged working time and decreased setting time, supposedly without decreasing the strength or increasing the solubility of the cement in the oral environment.

The rationale for employing such techniques may be based on the work of Henschel[1] who measured working and setting times of zinc phosphate cement mixed at temperatures ranging from 40 to 100°F. He found that cements mixed between 40-60°F showed increased working times as compared with those mixed at room temperature or higher. Jendresen[2] substantiated these results by demonstrating that the working time of zinc phosphate cement mixed at 45°F was more than double that of cement mixed at room temperature (70 ± 4°F). He also showed that lowered mixing temperatures result in a 30-50% decrease in setting time.

It was the purpose of this study to investigate and describe the effects of mixing zinc phosphate cement on both frozen (minus 10°C) or refrigerated (6°C) glass slabs. The specific objectives were to document changes in working time, setting time, diametral tensile strength, compressive strength, and solubility resulting from mixing on a cold slab. Such information would clearly demonstrate the effect of mixing over a wide temperature range on the properties of zinc phosphate cement.

METHODS AND MATERIALS

Three commercially available zinc phosphate cements were evaluated to describe their physical and mechanical properties after mixing on cold glass slabs. The cements are given in Table I.

All cements were mixed on glass slabs previously stored for at least 24 hours at either room temperature (23 ± 2°C), in a refrigerator (6 ± 2°C), or in the freezer compartment of a refrigerator (—10 ± 2°C). All slabs used for mixing at reduced tempera-

TABLE I

Cement	*Manufacturer*	*Batch #*
A. Zinc Cement	Stratford-Cookson Co.	112073
B. Zinc Cement Improved	S. S. White Co.	2057401
C. Fleck's Extraordinary Cement	Mizzy, Inc.	C 74

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tures were sealed in plastic bags during storage. Both the cement powder and liquid were stored at room temperature. All specimens were prepared in an environment of 23 ± 2°C and 50 ± 10 percent relative humidity.

No attempt was made to eliminate any moisture which may have condensed on a slab prior to or during the mixing procedure. Photographic records were made of the glass surfaces at various periods of time after mixing to document the amount of moisture condensation on the slab. The powder/liquid ratio for all specimens was based upon the necessary amount of powder added to 0.5 ml of liquid to produce a consistency of 26.5 ± 1 mm as described by Cameron.[3] Such a viscosity was used rather than the standard viscosity described in the A.D.A. specification for zinc phosphate cements (30 ± 1 mm) because it is the consistency used by practicing orthodontists.[3] Setting time for all cements was determined in accordance with A.D.A. Specification No. 8. The time of set was recorded as the amount of time from the beginning of mix until the needle of a one pound Gilmore needle no longer penetrated the surface of the cement.

Since no standard working time test for cements exists, setting time measurements were determined while the cement remained on the slab. Although such a method does not define the working time, it does give some measure of the comparative rates at which cements lose plasticity. In addition, two orthodontists and a dental assistant determined working times on a subjective basis. Working time was recorded as the period from the beginning of mix until the evaluator felt that the cement had reached a consistency beyond which further delay would prohibit proper seating of an orthodontic band. Three working-time determinations were made by each evaluator at each of the three mixing temperatures. Only one cement was evaluated for working time. Results were recorded to the nearest thirty seconds.

Both compressive and diametral tensile strengths were determined for all cements at the three mixing temperatures in accordance with A.D.A. Specification No. 8. Since cements are extremely brittle, conventional tensile testing techniques are difficult or impossible to employ. Therefore, to obtain tensile values, the diametral compression test was used. In this test a cylindrical specimen is placed on its side and loaded under a compressive force. Both strength values were determined using a universal testing machine. In addition, solubility and disintegration measurements were conducted on all brands of cement after mixing at the three different temperatures.

Results

Moisture condensation was evident on all refrigerated and frozen glass slabs. In all cases moisture condensation was in evidence at the beginning or at some time during the mixing procedure.

As the temperature of the mixing slab was decreased, the amount of powder necessary to maintain a constant viscosity increased. As can be seen (Fig. 1), all three proprietary cements reacted in a similar manner. Depending upon the brand of cement, the amount of powder necessary to achieve a cement mix of orthodontic consistency (26 ± 1 mm) when mixed on a refrigerated slab (6°C) was 49 to 70% greater than those mixed at room temperature. When mixed on frozen glass slabs (—10°C) the additional amounts of powder necessary to maintain the same viscosity were 63 to 91% more than those mixed on room temperature slabs.

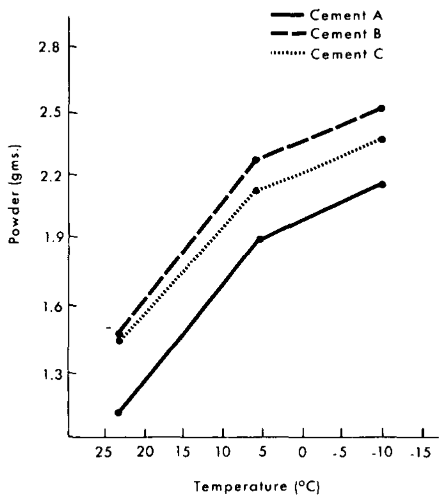

Fig. 1 Amount of powder necessary to achieve a standard consistency (26.5 ± 1 mm) compatible with orthodontic use. As the temperature is reduced, more powder incorporation is required.

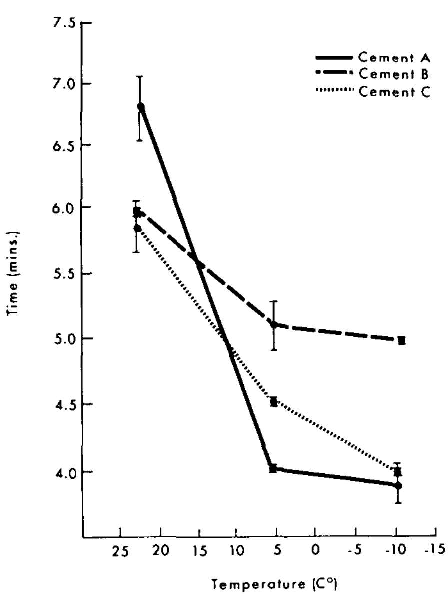

Fig. 2 Setting times of three proprietary cements in a simulated oral environment (37°C and 100% relative humidity) when mixed on slabs at the three indicated temperatures (23°C, 6°C, —10°C). The colder the mixing temperature, the faster the setting time when the cement is transferred to an oral environment.

Figure 2 shows the setting times for the various cements when mixed on glass slabs at three different temperatures and then transferred to a simulated oral environment (37°C and 100% relative humidity). As can be seen, all cements exhibited a decreased setting time when mixed on cooled glass slabs and then allowed to set in an oral environment. Decreases in the setting times ranged from one and a half to three minutes depending upon the cement. There was little difference in setting time between cements mixed on frozen glass slabs and those mixed on refrigerated slabs. A Student "t" test revealed a significant difference ($P < 0.05$) between the setting times of cements mixed on room temperature slabs and those mixed on surfaces at reduced temperatures.

Figure 3 portrays the setting-time determinations (working time) of cements allowed to remain on the mixing slab. Note that with one exception a proportional relationship existed between setting time and temperature of the glass slab on which the cements were mixed.

Working-time measurements as subjectively determined by three evaluators are demonstrated in Figure 4. Although the values for these working times were considerably less than the setting time values of cements allowed to remain on the slab at various temperatures, a similar relationship exists. That is, the rate of set and loss of plasticity is decreased as the temperature of the mixing slab is decreased. Mixing cements on a refrigerated slab increased the working time nearly three times whereas those mixed on a frozen slab remained in a manageable state for nearly seven times as long as compared with room-temperature mixes.

Results of the compressive strength tests indicated that with one exception there were no significant differences in

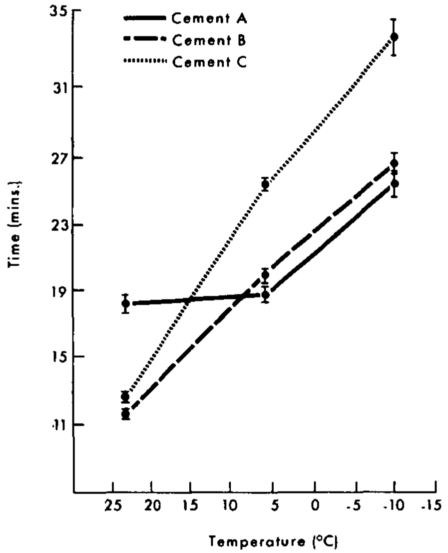

Fig. 3 Setting times of three proprietary cements (working time) when left on the respective mixing slab. Note that the colder the mixing temperature, the longer the working time.

values regardless of the slab temperatures on which the cements were mixed (Fig. 5). Similarly, there were no significant differences between diametral tensile values of cements mixed on slabs of varying temperatures (Fig. 6). Finally, solubility and distintegration values for all cements when mixed on slabs at all three temperatures were all within the limits defined by the specification for zinc phosphate cement. Again, no significant differences could be attributed to differences in slab temperatures.

Discussion

Results from the various tests conducted on zinc phosphate cement indicated that properties of strength as well as solubility and disintegration remained constant regardless of how much the temperature of the glass slab was reduced. Such results would at first seem unexpected since the amount of moisture condensation at subroom temperatures was abundant. Norman[4] has shown that moisture contamination re-

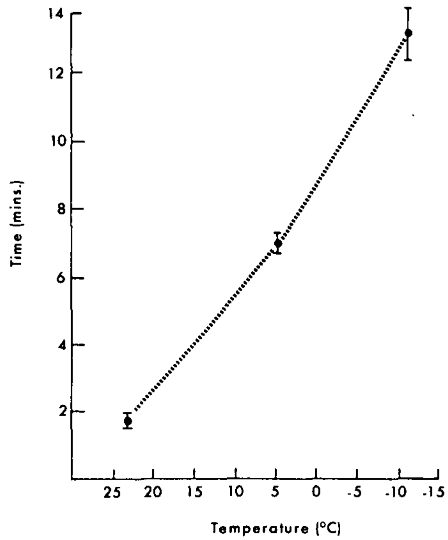

Fig. 4 Working time of Cement C as determined by three evaluators. Each data point represents the mean value for the three evaluators.

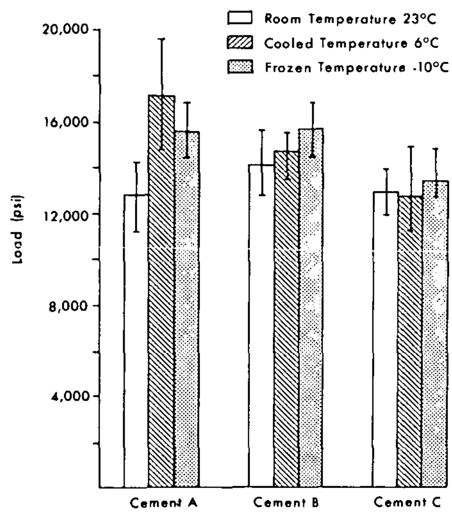

Fig. 5 Compressive strengths of three cements when mixed on glass slabs of different temperatures.

sults in an increased solubility as well as decreased compressive and tensile strengths. In fact, he has shown that a 10% increase in water content results in a 30% reduction in strength. Norman's tests, however, were conducted using a constant powder/liquid ratio.

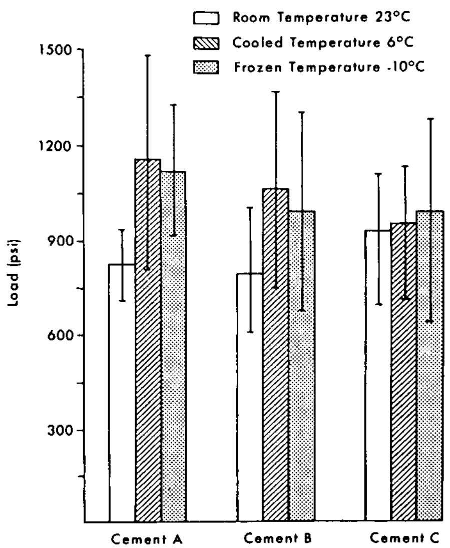

Fig. 6 Diametral tensile strength of three cements when mixed on glass slabs of different temperatures.

In this study the physical and mechanical properties were determined on cements in which the powder/liquid ratio was increased as the temperature was decreased to maintain a constant viscosity. Increasing the powder/liquid ratio, as was demonstrated by Paffenbarger,[5] results in improved mechanical characteristics. It is probable that the final result will depend upon which of these factors is more influential.

Although the absolute values for the setting-time determinations of cement left remaining on the glass slab (working time) were longer than the subjective working time measurements determined by three evaluators, values for both tests were generally proportional to temperature of the mixing slab and indicate that reducing the temperature of the mixing slab produces a cement mix which exhibits a much longer working time. The frozen slab technique offers an excellent means of extending the working time of zinc phosphate cement by as much as three and one half times without sacrificing strength or increasing solubility. By returning each loaded band to the frozen glass slab until all have been coated with cement, it is possible to provide the necessary working time that is so often insufficient in a clinical setting.

The setting-time measurements obtained in a simulated oral environment indicated that in general the greatest decrease in setting time occurred between room temperature and 6°C, with little or no change in setting time between 6 and —10°C. Clinically, this means that cements mixed on a cold slab to a specified consistency will be ready for scaling well before those mixed on a room temperature slab. This would allow scaling of excess cement to be accomplished sooner after the banding procedure.

Summary and Conclusions

A series of proprietary zinc phosphate cements were evaluated after mixing on glass slabs at three different temperatures, 23°, 6° and —10°C. Working time, setting time, diametral tensile strength, compressive strength and solubility were investigated. From the results of the tests the following conclusions were made:

1) As the temperature of the mixing slab was decreased, the amount of powder required to maintain a constant viscosity increased.

2) Compressive, tensile, and solubility values remained constant as the temperature of the mixing slab was decreased provided a consistent viscosity was maintained.

3) Mixing zinc phosphate cement substantially below the dew point is an acceptable practice provided the powder/liquid ratio is modified accordingly.

4) Although the setting time of zinc phosphate cement generally remained constant at temperatures below 6°C,

the working time continued to increase at —10°C.

5) Mixing zinc phosphate cement on a cold mixing slab increases the working time on the glass slab and decreases the setting time in an oral environment. Both of these alterations are clinically beneficial to the orthodontist.

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