The Effect of Mixing Method, Slab Temperature, and Humidity on the Properties of Zinc Phosphate and Zinc Silicophosphate Cement

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Although the bonding of brackets directly to tooth enamel has become common practice in orthodontics, the traditional cementing technique using bands and zinc phosphate cement will continue to be widely used. Thus, it is of real and practical benefit to the orthodontist to be aware of any improved techniques in the use of these cements which will enhance their convenience of manipulation or improve their physical and mechanical properties.

Several studies have been published which discuss the advantages of cold temperature mixing of zinc phosphate cement. Jendreson[1] has shown that the working time of cement mixed at 45°F is more than twice the working time of cement mixed at room temperature. In addition, he demonstrated that the reduced temperature mixing technique caused a 30-50 percent reduction in setting time in oral environment. Kendzior, Leinfelder, and Hershey[2] and Deal, Scholz and Jendreson[3] evaluated the properties of a series of zinc phosphate cements at both refrigerated and frozen temperatures and made similar observations. They found that as the temperature of the mixing slab was decreased, the amount of powder required to maintain a constant viscosity increased. Further, they noted that while reduced temperatures increased the working time on the glass slab and decreased the setting time in the mouth, strength properties and solubility remained relatively constant regardless of mixing slab temperature. These findings were later substantiated by Tuenge.[4]

A review of the above articles indicates that mixing zinc phosphate cement on a cold slab does indeed result in a cement which is not only more convenient to use, but also is comparable in terms of its physical and mechanical properties to cement mixed using traditional techniques. However, as is so often the case in reviewing reports of scientific investigations, the above articles raised a number of new, clinically relevant questions concerning the use of cold mix cements. These questions, listed below, were the target areas for the current investigation.

First, what are the effects of cold temperature mixing on silicophosphate cement as opposed to zinc phosphate cement? Secondly, what are the effects of mixing technique (rapid or slow incorporation of powder) on the behavior and properties of the cements? Thirdly, given the fact that moisture condensation is always present on the cold mixing slab, what are the effects of this moisture "contamination" and does cement mixed in an artificially desiccated environment exhibit improved properties? Finally, what are the effects of cold temperature mixing on the retentive properties of orthodontic bands cemented using this mixing technique?

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Methods and Materials

Three commercially available zinc phosphate and two silicophosphate cements were investigated (Table I). All cements were mixed on glass slabs previously stored for 24 hours at either room temperature (23 ± 2°C), in a refrigerator (6 ± 2°C), or in a freezer compartment (—10 ± 2°C). All slabs used for mixing at reduced temperature were stored in sealed plastic bags while the powder and liquid of all cements were stored at room temperature.

All cements were mixed at a constant environmental temperature of 23 ± 2°C and at a relative humidity of 50 ± 10 percent, except for the frozen slab condition which was also examined at a relative humidity of less than 10%. The environmental conditions were maintained by preparing the samples in a chamber originally designed as an infant incubator. While the system could readily be adjusted to maintain moderate to high levels of humidity, it was necessary to modify the chamber to create a low humidity environment by forcing compressed air through a chamber of dried calcium carbonate.

Consistency determinations were made for each of the cements prior to testing. The consistency test used in this study was based on that described by Cameron.[5] This standard provides a thicker viscosity than that normally used for cementing cast restorations and more closely resembles usual orthodontic consistency. Otherwise, the consistency test was the same as that described in A.D.A. Specification No. 8 for zinc phosphate cement.

Two methods of mixing the cements were evaluated. The first method consisted of incorporating the powder over a 90 second period and is the same as that described in A.D.A. Specification No. 8. In this technique small increments of powder are added to the liquid followed by increasingly larger portions. A total of 90 seconds is needed to complete the mixing procedure. This method will be referred to as the slow mix. The second method consisted of immediately incorporating all the powder into the liquid at one time and spatulation continued for a period of approximately 30 seconds. This method will be referred to as the rapid mix.

All physical and mechanical tests were carried out in accordance with the A.D.A. specification for dental zinc phosphate cement and/or those methods described by Kendzior, Leinfelder and Hershey.[3] Band retention measurements of the various cements were also

TABLE I

Cements Used in the Investigation.

Cement	*Letter Designation*	*Manufacturer*	*Batch #*	*Properties Tested*
Fleck's Extraordinary Zinc Phosphate	A	Mizzy Inc. Clifton Forge, Va.	C 79	Those in A.D.A. Specification No. 8 and Band Retention
Zinc Phosphate Improved	B	S. S. White Philadelphia, Pa.	1407210	Those in A.D.A. Specification No. 8 and Band Retention
Ames Zinc Phosphate	C	Zulauf Associates Lubbock, Texas	Test Sample	Consistency, Band Retention
Fluoro-Thin Silico-Phosphate	D	S. S. White Philadelphia, Pa.	447509	Those in A.D.A. Specification No. 8 and Band Retention
Kryptex Silico-Phosphate	E	S. S. White Philadelphia, Pa.	747405	Consistency, Band Retention

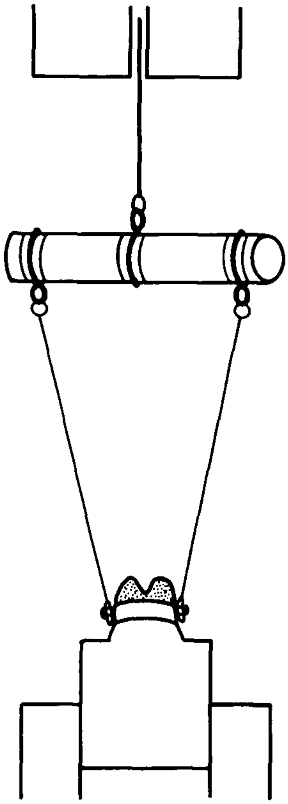

Fig. 1 Schematic illustration of the apparatus for testing the retentive strengths of orthodontic cements, as designed and described by Rich, Leinfelder, and Hershey.[6]

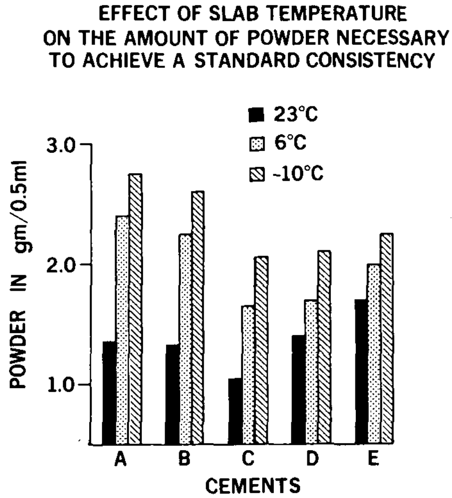

Fig. 2 The effect of mixing temperature on powder to liquid ratio. Note that as slab temperature decreases, more powder must be added to a given amount of liquid to maintain the standard consistency.

determined under the same conditions. The test for determining the force necessary to dislodge the bands was patterned after the methodology described by Rich, Leinfelder, and Hershey.[6] A schematic illustration of the device is shown in Figure 1. All five cements were used for determining standard consistencies and band retention. One silicophosphate (Fluoro-thin) and two zinc phosphate cements (Fleck's Extraordinary and Zinc Phosphate Improved) were evaluated for working and setting times, compressive and tensile strengths, and solubility.

Results

Results of the tests for consistency standardization are shown in Figure 2. Both zinc phosphate and silicophosphate cements required more powder to achieve a standard consistency when mixed on refrigerated or frozen slabs than when mixed on a room temperature slab. The increase was greater for the zinc phosphate than for silicophosphate cements. On the six degree Centigrade slab the percentage increase in additional powder ranged from 57 to 78 percent for the zinc phosphate cements and from 18 to 25 percent for the silicophosphate cements. On the minus ten degree slab the percentage increase ranged from 95 to 100 percent for the zinc phosphate cement and from 32 to 50 percent for the silicophosphate cement. These results were obtained by mixing the cements in accordance with A.D.A. Specification No. 8. When all the powder was immediately incorporated into the liquid, the following results were obtained. On room temperature slabs, the rapid mixing technique re-

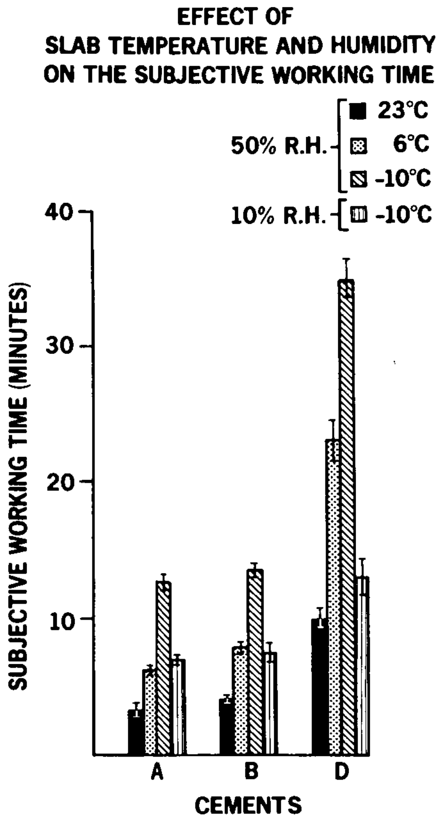

Fig. 3 Changes in working time on the mixing slab produced by alteration of temperature and relative humidity. Working time increased as the temperature of the slab was lowered with the silicophosphate cement (D) showing the greatest increase. Lowering the humidity markedly reduced working time.

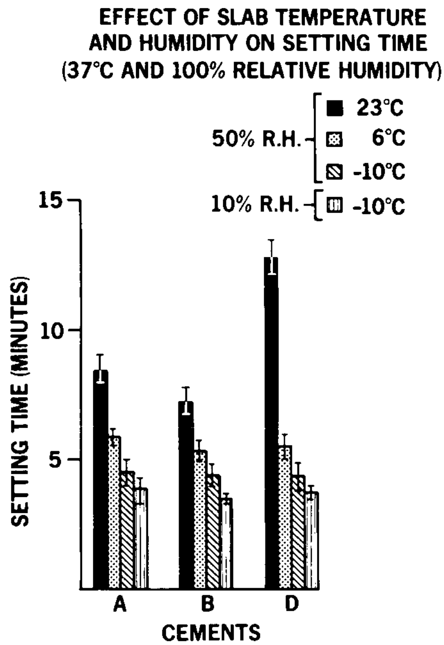

Fig. 4 Changes in setting time in simulated oral environment produced by alteration of temperature and relative humidity; the colder the slab, the faster the setting time in an oral environment.

sulted in decreased powder/liquid ratios. This decrease was 17 to 28 percent for zinc phosphate cement and seven to ten percent for silicophosphate cement. At refrigerated or frozen temperatures, the powder/liquid ratio was the same regardless of the type of cement or rate at which the powder was incorporated into the liquid.

Working time measurements were subjectively determined by three independent evaluators and are illustrated in Figure 3. As the slab temperature was decreased, the working time for both types of cements was increased. Depending upon the temperature of the slab, the working time for zinc phosphate ranged from three to four minutes to about thirteen minutes. The working time for silicophosphate cement under the same conditions ranged from 10 to 35 minutes. At subroom temperatures the rate of powder incorporation had no effect on working time. At room temperature, however, the rapid mix cements exhibited less working times than their conventionally mixed counterparts. Mixing on frozen slabs in the absence of moisture routinely resulted in shorter working times. As can be seen in Figure 3, the silicophosphate cement was the most affected by the absence of moisture on the frozen slab.

The mean setting times for the various cements in a simulated oral envi-

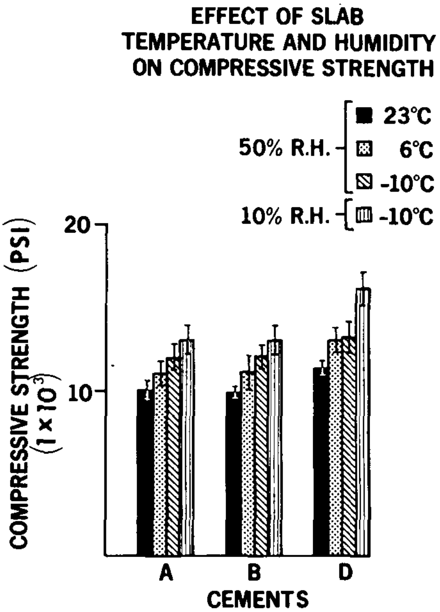

Fig. 5 Compressive strength of cements mixed at different temperatures and relative humidity. As slab temperatures decreased there were slight to moderate increases in compressive strength values.

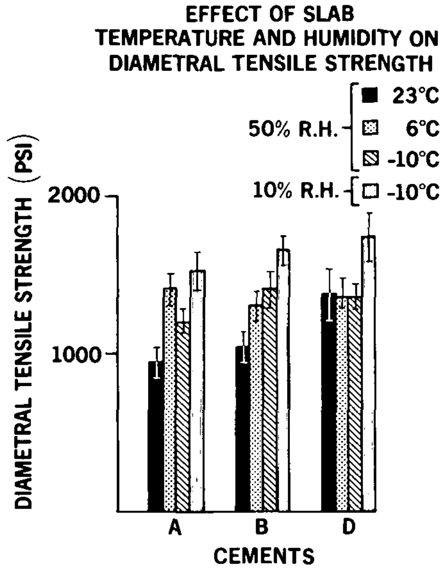

Fig. 6 Diametral tensile strength at different mixing temperatures and relative humidity.

ronment are illustrated in Figure 4. In general, as the slab temperature was decreased, the setting time also decreased. Of the two types of cements evaluated, the silicophosphate was more affected. The rate at which the powder was incorporated into the liquid had no effect on any of the cements mixed at subroom temperatures. At room temperature, however, rapid mixing caused a decrease in the setting time of zinc phosphate as well as silicophosphate. Mixing in the absence of moisture had no significant effect on the setting time of either type of cement.

Slight to moderate increases in compressive strength values were observed for both types of cements as the slab temperatures were decreased (Fig. 5). Furthermore, none of the strength values for any of the cements mixed at subroom temperatures were affected by the manner in which they were mixed. At room temperature, however, both zinc phosphate and silicophosphate cements exhibited lower strength values when prepared by the rapid mix technique. For example, under this condition, zinc phosphate cement experienced nearly a 20 percent strength decrease while strength of the silicophosphate cement decreased 6 percent. When mixed at low temperatures, cements mixed in the absence of moisture had increased compressive strength values as compared with cements mixed in the presence of moisture. This increase was 10 percent for zinc phosphate and 20 percent for silicophosphate.

When tested under the same conditions, the diametral tensile strength (Fig. 6) values followed a trend similar to those for compressive strength. Diametral strength increased as the mixing slab temperature decreased. The rapid incorporation of powder into the liquid caused a reduction in tensile strength by 25 percent when mixed at room temperatures. However, at lowered

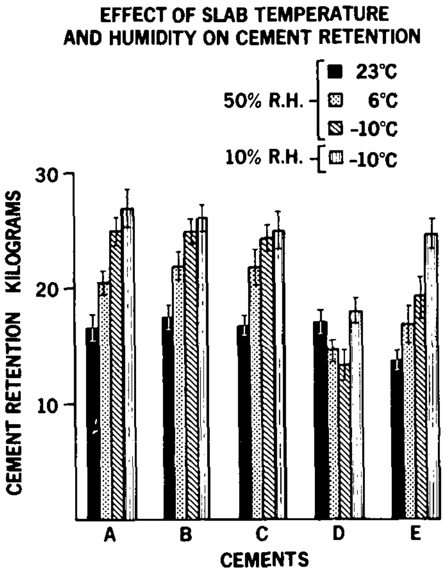

Fig. 7 Amount of force required to dislodge cemented bands as determined by the Instron. Generally, lowered slab temperatures resulted in more force required to remove the band.

temperatures there was no difference in the tensile strength of the slow or rapid mix cements. Tensile strength of zinc phosphate increased 15 to 20 percent and that of silicophosphate increased 25 percent.

Band retention measurements for the various cements and mixing temperatures are illustrated in Figure 7. Under room temperature mixing conditions the values for all cements were nearly equal. Except for one silicophosphate cement (Fluoro-thin), reducing the slab temperatures resulted in higher band retention values. Lack of moisture contamination significantly enhanced the band retentive characteristics of silicophosphate cement but had little or no effect on the zinc phosphate cements.

The mean solubility and disintegration values for all cements at conditions tested were within acceptable limits as defined by A.D.A. Specification No. 8.

Discussion

To maintain a constant viscosity of the cement mix it was necessary to increase the powder/liquid ratios for both types of cement when mixing at low temperatures. The extra powder was necessitated by the dilution brought about by moisture condensation when the temperature of the mixing surface is reduced below the dew point. Doubling the amount of powder at reduced temperatures to maintain a constant viscosity is not unrealistic when the amount of moisture incorporated is considered. It was determined in this study that the average volume of water incorporated into a 10 to 12 drop mix of zinc phosphate cement on a frozen slab is about 0.12 grams or between 2 and 3 drops. This was done by placing refrigerated slabs in the controlled humidity environment and noting change in weight as the condensation formed.

Working time values for all cements tested were substantially lengthened at lowered temperatures. An inversely proportional relationship generally existed between working time and temperature of the mixing slab. Increased working times can be related to a low level of chemical reaction at lowered temperatures. At the molecular level it is felt that, although the powder goes into solution at lowered temperatures, the process by which the cement hardens (nucleation and crystal growth) is greatly retarded. In this study, once the temperature of the cement was elevated to that of a simulated oral environment, the hardening process proceeded rapidly. Although the absence of moisture at reduced temperatures resulted in decreased working times, it had less effect on the setting time at oral temperatures. It is probable that the presence of moisture at lowered temperatures merely serves to prolong the period of plasticity of the cement.

The compressive and diametral tensile strength values observed in this study are in agreement with those of Jendreson and Kendzior, Leinfelder, and Hershey. Relatively constant values were maintained regardless of the temperatures at which they were mixed. It is probable that potentially higher values at reduced temperatures would have been realized if moisture contamination had been eliminated. Apparently the effect of moisture contamination offset the effect of increased powder/liquid ratios. Evidence for this is the observation that the diametral tensile strengths of both types of cements were substantially higher when mixed on a moisture free surface.

Although the rate of incorporation of powder at reduced temperatures had little effect on the properties tested, rapid mixing on room temperature slabs generally resulted in less than optimum properties. At room temperature small increments are slowly added to the liquid and thoroughly spatulated to retard the heat of reaction. Large incremental additions at room temperature cause a high evolution of heat. Consequently, not only will the rate of set be increased, but also a reduced powder/liquid ratio will result. At reduced temperatures the kinetics of reaction are sufficiently lowered so that as long as the particles are completely wetted, the technique of mixing makes little difference.

Except for one of the silicophosphate cements, reducing the mixing temperature generally resulted in improvement in band retention. Furthermore, while retention values for zinc phosphate cement remained relatively constant when mixed on moisture free slabs, those for silicophosphate cement increased. Band retention values for zinc phosphate are in good agreement with those reported by Rich, Leinfelder, and Hershey but somewhat lower than those reported by Houston and Miller.[7]

In clinical use, cold temperature mixing will provide the clinician with a cement with a substantially longer working time on the slab and a substantially shorter setting time in the oral environment. Thus, more bands can be cemented with a single mix of cement and less time need be allowed for the cement to set before archwires are placed. Because the powder can be incorporated immediately, mixing requires substantially less time. In addition, this allows premeasured portions of liquid and powder to be mixed, further reducing time required for preparation of the cement. The clinician can utilize this mixing technique with the knowledge that the physical and mechanical properties of the cement are probably superior to conventionally mixed cement and that orthodontic bands cemented with cold mix cement are at least as retentive as bands cemented with traditionally mixed cement.

Summary and Conclusions

A series of zinc phosphate and silicophosphate cements mixed at room temperature (23°C), refrigerated temperature (6°C) and frozen temperature (—10°C) were evaluated for compressive and diametral tensile strength, working time, setting time, solubility and band retention. In addition, the effect of the rate of incorporation of powder into the liquid and the effect of moisture contamination on the physical and mechanical properties of the cement were investigated.

From the results of this study the following conclusions were made:

1) As the temperature of the mixing slab decreased, the powder/liquid ratio necessary to achieve a standard consistency increased.

2) Working time on the mixing slab substantially increased as the mixing temperature was lowered for both types of cement.

3) Setting time in a simulated oral environment was substantially decreased for both types of cement mixed at reduced temperatures.

4) Lowered mixing temperature generally resulted in slight to moderate increases in compressive and tensile strengths for the zinc phosphate cements. The silicophosphate cement was less affected under the same conditions.

5) At reduced temperatures the rate of powder incorporation had no effect on any of the properties tested. At room temperature rapid incorporation resulted in less than optimum properties.

6) At reduced temperatures, moisture-free mix generally resulted in higher compressive and tensile strengths for both types of cement. Moisture-free mixes at subroom temperatures also resulted in reduced working times.

7) Mixing zinc phosphate cement on a frozen slab significantly increased band retention.

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