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RHEOLOGY OF LUTING CEMENTS,(U)
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A rheometer has been developed to simulate the extrusion of luting cement from beneath a full crown during seating using clinically realistic shear rates. Five luting cements were studied and differences in rheological behavior were illustrated. These measurements demonstrate the importance of consideration of the effects of shear rate on viscosity when evaluating a cement for optimal clinical utilization.

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Rheology of Luting Cements

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Paper presented at International Association for Dental Research Meeting, Washington, D.C., 1978.

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Synopsis

A rheometer has been developed to simulate the extrusion of luting cement from beneath a full crown during seating using clinically realistic shear rates. Five luting cements were studied and differences in rheological behavior were illustrated. These measurements demonstrate the importance of consideration of the effects of shear rate on viscosity when evaluating a cement for optimal clinical utilization.

Key Words

Rheology
Viscosity
Consistency
Luting cement
Newtonian liquid
Pseudoplastic liquid

Introduction

Knowledge of the apparent or "working" viscosity of dental luting cements is of great importance to the clinician. Apparent viscosity is the viscosity or relative consistency of a material at a particular shear rate. A fluid whose viscosity remains constant whatever the shear rate is known as a Newtonian liquid. One whose viscosity decreases as the shear rate increases is called pseudoplastic; one whose viscosity increases with increased shear rate is known as dilatant.

With knowledge of the behavior of any particular luting cement to various rates of shear, the clinician could vary his cementing technique. For example, if he is using a type of cement whose viscosity decreases with increasing shear rates then the use of rapid quick tapping forces would encourage the extrusion of the luting cement from beneath the casting. If the cement viscosity increases with high shear rates it would be more effectively extruded by a slow steady application of force.

A variety of tests have been used to examine the rheology of dental cements. Friend[1] simply used subjective evaluations by clinicians ranking mixes as too thick, too thin, or just right. Such an evaluation would not provide information on the relative behavior of different cements during cementation since one with pseudoplastic characteristics might appear thick but in reality be more readily extruded than a Newtonian liquid of comparable subjective thickness. The ADA test for establishing "testing consistency" and also film thickness makes use of a parallel plate plastimeter.[2] A number of investigators have used the film thickness measured at various time intervals after mixing, under various environmental conditions, and with different powder/liquid ratios for evaluating consistency.[3-6] Batchelor and Wilson[7] stated that the parallel plate plastimeter does not measure the rate of flow but measures a static equilibrium, where the applied force is balanced by the internal shear stress. For this reason the parallel plastimeter falls short as an instrument for examining the rheology of different types of cement.

Plant, et al[8] compared the setting characteristics of various cements via traces obtained from the oscillating rheometer. Differences were observed in the patterns of change in fluidity of setting cements as related to the type of cement, temperature and powder/liquid ratios.

Vermilyea, Powers and Craig[9] used a rotational viscosimeter to measure the viscosity of zinc phosphate and polycarboxylate cements during setting. They modified the spindles of the apparatus so that small amounts of liquid could be used. The spindles were recalibrated using viscosity standards. They observed some change in viscosity with increasing shear rates, but found temperature to have a greater effect. However a basic limitation of the technique is the design of the rotational viscosimeter. Most commercial rotational viscosimeters operate at shear rates below 20 second^{-1}. Since the tapping closure rate of a normal human mandible can surpass 8000 mm/min[10], it can be inferred that the shear rates present at the margins of castings during cementation may be higher by several orders of magnitude than those produced in the rotational viscometer. A highly non-Newtonian cement might behave quite differently during cementation of a casting than the data from a rotational viscosimeter would indicate.

The purpose of this study was to design and test an instrumenf for investigation of the viscous behavior of cements as a function of rate and time of application of the seating force.

Methods and Materials

Instrumentation

A diagram of the apparatus designed and fabricated for investigation of the rheological behavior of cements during seating of a casting is shown in Figure 1. The instrument essentially is a ram and piston penetrometer. The reservoir capacity of 1.25 ml was selected because it was possible to mix the dental cement in amounts of 1 ml by the accepted clinical technics. The floor of the reservoir was formed by a threaded plug to permit removal and hence facilitate cleaning of the apparatus. There was a 30 micron circumferential clearance between the piston and walls of the reservoir. A gap of this magnitude was felt to represent a realistic marginal opening through which cement would be extruded during seating of a cast full crown.

The piston was driven into the cement reservoir by means of a universal testing machine* at a fixed rate of travel, and the required force was recorded. The range of head speeds was established in a series of preliminary tests. With a head speed of 1.25 mm/min the maximum travel time was 8 minutes, and this permitted the entire setting cycle to be monitored. The highest head speed at which strain behavior of the cements could be monitored was 80 mm/min. The cements were tested at head speeds of 1.25, 2.5, 5, 10, 20, 40 and 80 mm/min. It was felt that the data obtained over the range of shear rates was sufficient

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* Instron Corporation, Canton, MA.

to allow reliable inferences to be made with respect to rheological behavior of the cements.

In order to determine approximately the magnitude of shear rates developed in the apparatus, preliminary tests were conducted with silicone polymer viscosity standards of 12,000, 57,000 and 105,000 centipoise.* The resistance curves obtained as related to head speeds are presented in Figure 2. The silicone polymers are Newtonian fluids at shear rates below 2000 second^{-1} but become pseudoplastic at shear rates in excess of 2000 second^{-1}.[11] Since the behavior of these silicone polymers was pseudoplastic in these tests the inference was that shear rates in excess of 2000 second^{-1} were produced in fluids when tested in this apparatus. A calculation of the shear stress developed in this device based upon chamber dimensions and the circumferential gap indicated that a shear rate of 2000 second^{-1} would be reached at a head speed of 7.6 mm/min.

Five cements were included in the study. They were: (1) a representative zinc phosphate cement; (2) a conventional zinc polycarboxylate; (3) a zinc silicophosphate; (4) a glass ionomer, formulated as a luting agent; and (5) a water mixed polycarboxylate. The materials, powder/liquid ratios and the film thicknesses obtained at those ratios are listed in Table I. The powder/liquid ratios employed for all materials except the zinc phosphate are those recommended by the respective manufacturers. The zinc phosphate cement mix at the recommended ratio was judged to be too viscous for clinical cementation, there-

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* Brookfield Engineering Laboratories, Stoughton, MA.

fore a somewhat lower powder/liquid ratio was used. The powders were proportioned by weight on an analytical balance and, with the exception of the conventional polycarboxylate, the liquids from bulk containers using glass tuberculin syringes. The manufacturer's plastic syringe was used for the polycarboxylate liquid.

The zinc phosphate and silicophosphate cement were mixed on cool glass slabs. The mixing slab was placed in a container of crushed ice for 2.5 minutes, then it was removed, dried and the powder and liquid dispensed onto it. Mixing was initiated promptly at 3 minutes from the time of removal of the slab from the ice. This procedure produced a consistent mixing slab temperature (61.7 ± .9^{o} F).

The other three cements were mixed on room temperature slabs since the manufacturers did not recommend cool slabs. In order to determine the effect of slab temperature on the rheological behavior of the conventional polycarboxylate cement a second series of tests was run on the material in which mixing was done on a chilled slab.

The manufacturers' directions were followed with all cements with respect to rate of addition of the powder, mixing procedure and time. Immediately after mixing, the cement was placed in the reservoir of the test apparatus, and 30 seconds after completion of the mix the test was initiated.

Prior to each test, a run was made with the apparatus empty in order to record the spring constant which was deducted from the subsequent readings. Also, as a control measure, the temperature of the test apparatus was monitored prior to loading with the cements and during the test runs.

Results

The resistance values for the zinc phosphate cement at the different crosshead speeds are shown in Figure 3. The increase in resistance is generally proportional to the increase in strain rate. When the resistance values at one minute after mixing are plotted against the strain rates (Figure 4) the resulting data points can be fit to a straight line with a correlation coefficient of .987. The behavior of this zinc phosphate cement can be described as Newtonian. To investigate the possibility that the passage of the piston through the cement was affecting the rate of setting and thus the resistance values, runs were made at 10 mm/min at successive time periods. If the testing process was affecting the set of the cement, the resistance values at the end of one run should not correlate with the values at the start of the next run. Since the three resistance curves are fairly contiguous (Figure 5),it can be inferred that the testing procedure did not markedly affect the setting of the cement.

The resistance values for the conventional zinc polycarboxylate cement at different crosshead speeds are shown in Figure 6. Replotting the resistance values obtained one minute after mixing against the strain rates (Figure 7) produces a curve suggestive of the behavior of a pseudoplastic material.

The resistance of polycarboxylate cement mixed on a chilled slab is compared with the resistance of the same cement mixed on a room temperature slab in Figure 8. Initially the consistency of the cement mixed on the cool slab is not less than the cement mixed at room temperature, but the cement mixed on the cooled slab maintains a lower consistency for a longer time.

The resistance curves for the zinc silicophosphate, glass ionomer and water mixed polycarboxylate cement tested at a head speed of 1.25 mm per minute are compared to those obtained for zinc phosphate and the conventional zinc polycarboxylate cement in Figure 9. At this low shear rate the glass ionomer cement shows more resistance and has a much shorter working time than the other cements. The working time for the water mixed polycarboxylate is considerably less than the conventional polycarboxylate and it affords more resistance to the piston. It is interesting that with this slow rate of shear the silicophosphate cement offers less resistance and has a longer working time than does the zinc phosphate cement.

Discussion

The data obtained for the silicone polymer viscosity standards in this test apparatus designed to simulate seating of a cast full crown indicate shear rates well in excess of 2000 second^{-1} would be developed in the fluid cement during cementation. In fact calculations indicate that shear rates of this magnitude were achieved when the travel of the piston into the reservoir was less than 10 mm/min. However, even the maximum speed used here of 80 mm/min is slow indeed, when it is considered that the closure rate of the human mandible can be more than 8000 mm/min. Thus it is apparent that during seating of a casting very high shear rates can be developed in the cement by tapping the casting into place or allowing the patient to seat the casting by rapid closure of the mandible. Shear rates of these magnitudes could easily induce pseudoplastic behavior in a shear labile luting agent.

The differences in the rheological behavior of zinc phosphate cement and polycarboxylate cement when subjected to increasing strain rates is most interesting. The zinc phosphate cements behaved in a Newtonian fashion over the entire range of strain rates employed here, while the conventional polycarboxylate behaved as a Newtonian liquid only at low strain rates. At higher strain rates it exhibited pseudoplastic properties. It is speculated that the pseudoplastic behavior of this cement is due to the presence of a polymer, since certain polymeric materials are known to behave as Newtonian liquids at low strain rates but are shear labile, and behave pseudoplastically at higher rates.

The apparent "viscous consistency" of the mix of polycarboxylate cement at the recommended powder/liquid ratio as compared to the consistency of the mix of the more familiar zinc phosphate cement has been difficult for the practitioner to accept. However, these data would indicate that because of the pseudoplastic behavior of the polycarboxylate cement the resistance to seating of the casting is no greater and perhaps less than when zinc phosphate cement is used. As was suggested previously, from a practical standpoint it would appear that seating of the casting with zinc phosphate or any cement that behaves in a Newtonian fashion could best be accomplished by applying force at a slow steady rate. Any sort of impact load would be contraindicated since the resistance would be increased proportionally to the rate of loading. On the other hand rapid loading, such as tapping, might be advantageous when seating a casting with a pseudoplastic cement, i.e. polycarboxylate cement, since resistance does not increase proportionally with the increasing shear rate.

With respect to mixing of the polycarboxylate cement the data indicate that mixing on a chilled slab does not reduce the initial viscosity of the cement but that it does extend working time. In many instances the longer working time would be clinically advantageous.

The zinc silicophosphate, glass ionomer and water mixed polycarboxylate cements were tested only at the lowest strain rate. Although some interesting comparisons might be made with respect to relative working properties of the materials the data for zinc phosphate and polycarboxylate indicate the fallacy of attempting to do so until tests over a range of strain rates have been conducted.

In view of Wilson and Batchelor's statement regarding the inability of the conventional film thickness test to predict working properties of cements, it is interesting to compare the resistance of the various cements at early times, i.e. 3 minutes (Figure 9) to the film thickness date (Table I). The water mixed polycarboxylate ranked fourth highest in resistance to seating of the piston at this low strain rate but was lowest in film thickness. However, if one removes this cement from the group there appears to be a reasonably good correlation between film thickness and the shear resistance of the other 4 cements. The zinc silicophosphate cement had the lowest film thickness and exhibited the lowest shear stress while the glass ionomer had the highest film and afforded greatest resistance to seating. The other 2 cements fell between the extremes both with respect to film thickness and resistance.

Conclusions

In conclusion, a new technique has been developed to measure the flow properties of dental cements during setting using clinically realistic shear rates. These measurements have demonstrated the importance of consideration of the shear rate dependence of the viscosity of this class of dental materials in making a clinical evaluation of their optimal utilization.

The authors wish to acknowledge Dr. A. P. G. Giorgini, Associate Professor, School of Civil Engineering, Purdue University, Lafayette, Indiana, for his aid in calculating the shear stress developed by the test instrument.

Commercial materials and equipment are identified in this report to specify the investigative procedure. Such identification does not imply recommendation or endorsement, or that the materials and equipment are necessarily the best available for the purpose. Furthermore, the opinions expressed herein are those of the authors and are not to be construed as those of the Army Medical Department.

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TABLE I

		Film Thickness Range	P/L Ratio
Zinc phosphate	Fleck's Cement Mizzy, Inc.	28 - 32	1.6cc/.6cc
Polycarboxylate (conventional)	Durelon Premier Dental Prod.	24 - 26	1.74/1.25
Silicophosphate	Fluoro-thin S. S. White Co.	24 - 28	1.4/.6
Glass ionomer	Fuji Ionomer Type 1 for cementation G-C Dental Industrial Corp.	32 - 36	1.6/1.0
Polycarboxylate (water-mixed)	Unident Sankin Corp.	16 - 24	2.85/.75

Captions

Figure 1. Diagram of rheometer designed to simulate extrusion of cement during seating of a full crown.

Figure 2. Resistance curves obtained for silicone polymer viscosity standards using the test apparatus with various cross head speeds.

Figure 3. Resistance curves for zinc phosphate cement when tested at various cross head speeds.

Figure 4. Resistance of zinc phosphate cement one minute after completion of the mix as related to cross head speed.

Figure 5. Resistance curves obtained for mixes of zinc phosphate cement when the tests were initiated at 1, 1.8 and 3 minutes after completion of the mix.

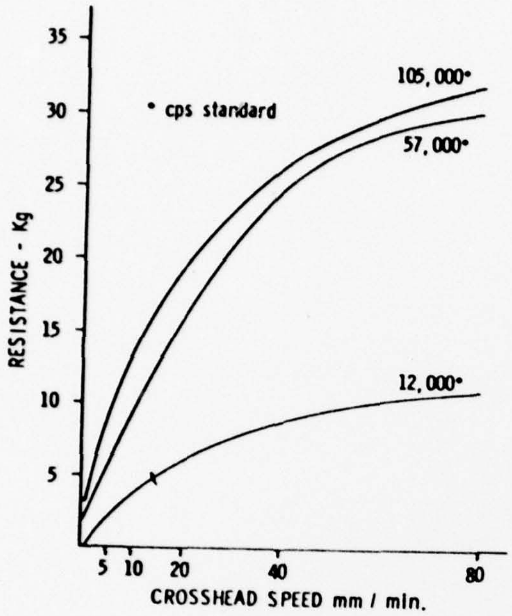
Figure 6. Resistance curves for polycarboxylate cement when tested at various cross head speeds.

Figure 7. Resistance of polycarboxylate cement one minute after completion of the mix as related to strain rate.

Figure 8. Resistance curves for polycarboxylate cement mixed on a chilled and on a room temperature slab.

Figure 9. Resistance curves for various cements when tested at a cross head speed of 1.25 mm/min.

VISCOSITY STANDARDS AS MEASURED IN TEST APPARATUS



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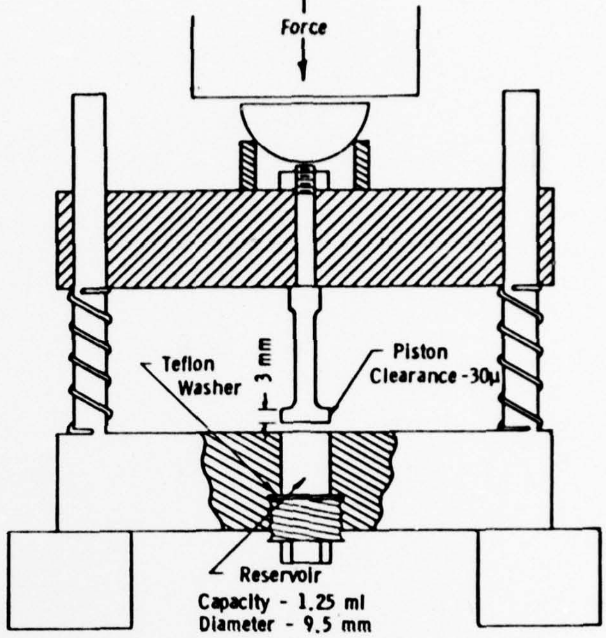
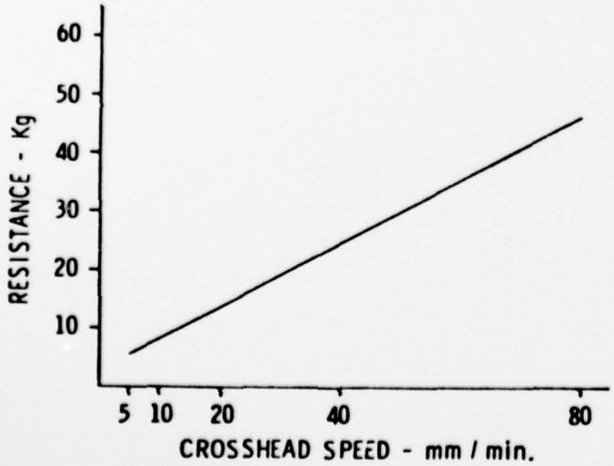


Fig 1

ZINC PHOSPHATE CEMENT
Resistance Related To Crosshead Speed
(one minute after mixing)



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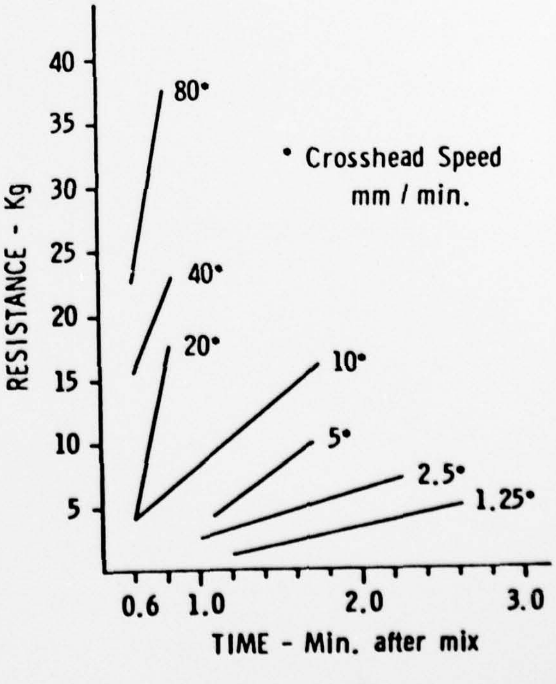
ZINC PHOSPHATE CEMENT
At Various Crosshead Speeds



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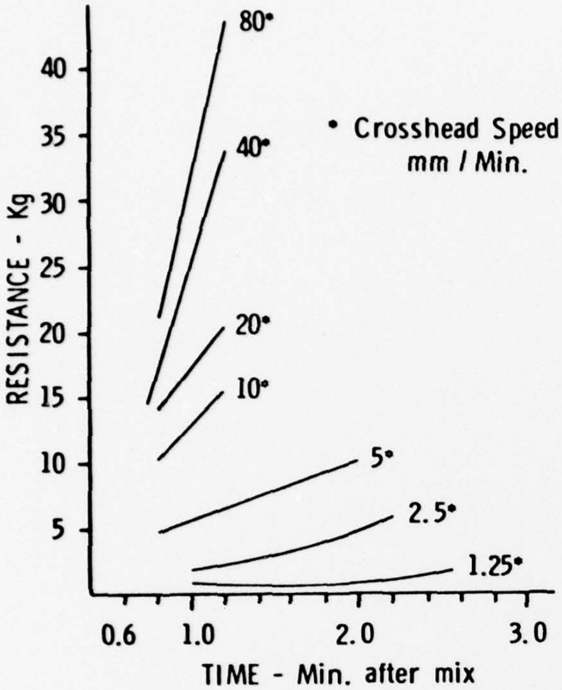
POLYCARBOXYLATE CEMENT
At Various Crosshead Speeds



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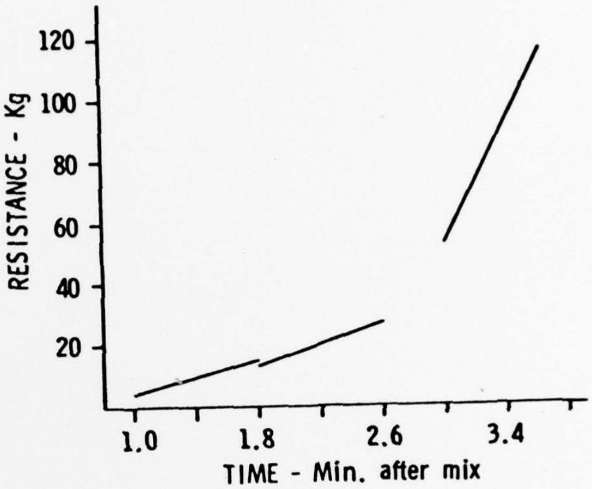
ZINC PHOSPHATE CEMENT
10 mm / min. Crosshead Speed Serial Runs



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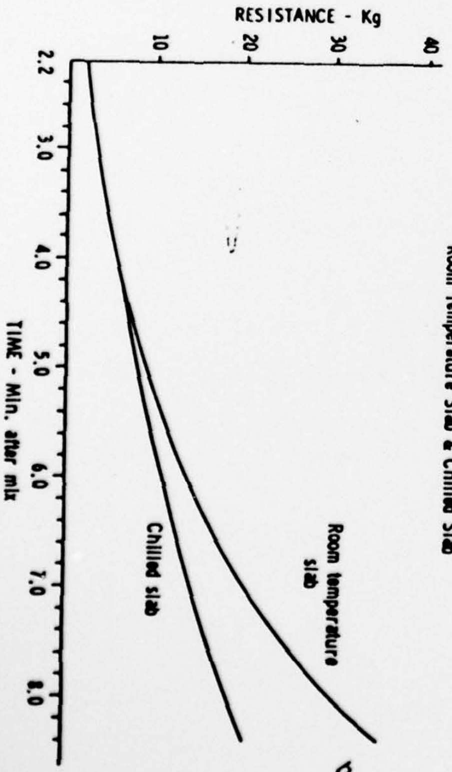

COMPARISON OF POLYCARBOXYLATE CEMENT
At 1.25 mm / min. Crosshead Speed
Room Temperature Slab & Chilled Slab

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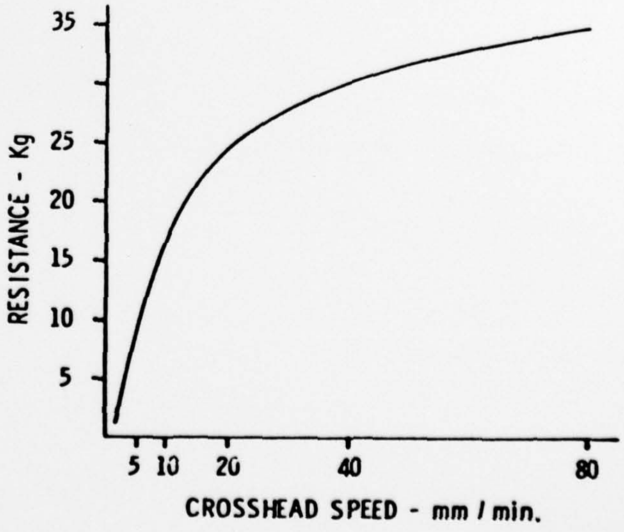
POLYCARBOXYLATE CEMENT
Resistance Related To Crosshead Speed
(one minute after mixing)



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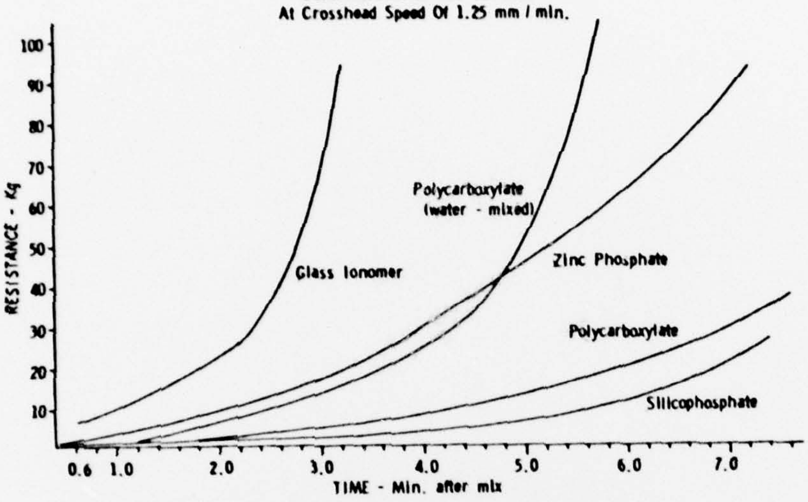

COMPARISON OF ALL CEMENTS
At Crosshead Speed Of 1.25 mm / min.