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VACUUM-MIXING OF SILICATE CEMENT

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INTRODUCTION

A silicate cement mixed by ordinary spatulation on a glass slab will — like most other highly viscous mixtures of dry powder and liquid — contain air bubbles of varying size. The bubbles are relatively small, frequently up to 50μ , rarely more than 100μ , and are usually shaped like irregular spheres. As a rule, the air bubbles cannot be seen with the naked eye, but are readily observed under the microscope at moderate magnification.

If the cement is mixed in a vacuum the total volume of air bubbles is visibly reduced, at the same time as the individual bubbles become smaller in size.

The purpose of this work is to show to what degree the porosity of a silicate cement is reduced by mixing under vacuum, and what influence such a reduction has upon selected properties of the material.

Lyon & Cosca (1967, 1968), in a study of vacuum-mixed silicate cements, were the first to demonstrate that vacuum-mixing reduced the porosity of the cement. They further stated that vacuum-mixed cement has a shorter setting time, is more translucent, and less soluble in water, citric acid, and artificial saliva.

MATERIALS AND METHODS

The test specimens were prepared from Silicap powder (batch No. b.057) and Silicap liquid (batch No. 661D) supplied by Etablissement Vivadent,

Received for publication, March, 27, 1969.

Schaan, Liechtenstein. This cement is moderately fast setting (the setting time according to F.D.I. standard specification No. 5 for dental silicate cement is seven minutes) and otherwise complies with the requirements of the specification. It has standard consistency when mixed in the ratio of 0.4 ml of liquid to 1.4 g of powder.

1. *Vacuum technic.* The containers used for mixing under vacuum were cylindrical glass tubes of 12 mm inside diameter. They were closed at the ends with rubber stoppers so that lumen of the tubes became about 20 mm in length. A weighed amount of powder was placed in the tube, which was then closed and mounted in the abovementioned manufacturer's mechanical silicate cement mixer (Silamat).

Addition of liquid and evacuation of the air in the glass tube took place in two different ways. Method A started with injection of a measured amount of liquid through one of the rubber stoppers by means of a precision syringe (accuracy ± 0.001 ml). Immediately after the injection the air was evacuated through a No. 8 hypodermic needle connected to a high vacuum source, whereby the pressure inside the glass tube was brought down to 2 Torr within 5 seconds. As soon as this had been accomplished the cement was mixed for 10 seconds. After mixing the glass tube was transferred to an automatic vibrator and vibrated a few seconds to consolidate the cement mix. Only then was the vacuum broken and the tube opened. The total time from the first contact between powder and liquid until removal of the stoppers was from 25—30 seconds. Method B was identical with method A, except that the glass tube was evacuated before introduction of the liquid.

Examinations of the cement mix obtained with method A showed a slight to moderate reduction in porosity, while a considerable reduction was achieved by using method B. So the cement in the following investigation was treated exclusively by method B.

Each mix was made up of approximately the same amount of powder and liquid as required for a large clinical filling. The amount of liquid was held constant at 0.057 ml, but the amount of powder was varied so as to produce standard consistency (200 mg) in some tests, while in other tests the powder content was decreased 10 % or increased either 10 % or 15 %.

The room temperature during vacuum-mixing as well as during the conventional mixing procedures (see points 2 and 3) was $22.0 \pm 1.0^\circ\text{C}$.

2. *Mechanical mixing without vacuum.* The mixing was performed as previously described under 1, but with normal atmospheric pressure in the glass tubes. The liquid was added by injection through one of the rubber stoppers of the glass tube. The mechanical mixing, with or without vacuum, accelerated the setting of the cement moderately, corresponding to a reduc-

tion in its setting time from 7 minutes to 5 minutes. The acceleration is due to an appreciable rise in temperature during mixing.

3. *Hand spatulation.* The mix was spatulated in accordance with the F.D.I. specification No. 5 for dental silicate cement.

4. *Measurement of porosity.* When freshly mixed cement is compressed into a thin disk between two glass plates a great number of air bubbles are caught on the boundary between cement and glass plate. As a result, the cement will appear more porous under the microscope than it actually is. Therefore the glass plate method cannot be used for a quantitative determination of cement porosity.

The procedure adopted instead was measurement of the specific gravity of the set cement by means of Archimedes' principle (weighing of the cement in air with 100 % humidity and in water), combined with a direct measurement of the porosity on polished sections by means of the point counter method as described in the following.

Five standard vacuum-mixes were stored 24 hours at 37°C in the sealed glass tubes in which mixing had taken place. The cement specimens were then removed from the tubes, and after measurement of their specific gravity they were ground flat and polished. On each section 200 points were examined, and in each specimen three sections at intervals of 0.2 mm were examined. Thus the points totalled 3000. The mean specific gravity for the five specimens was 2.204 (lowest value 2.199, highest value 2.210). The mean porosity according to the point counter method was 1.20 % (lowest value for one specimen 0.83 %, highest value 1.67 %). Calculated from these figures the specific gravity of the nonporous cement was 2.231 (lowest value 2.221, highest value 2.243).

Next, the porosity was determined for cement mixed mechanically without vacuum and for hand-spatulated cement. In the first case, the mixed cement was left 24 hours at 37°C in the closed glass tube; in the second case, the spatulated cement was at once transferred to a mixing glass tube and stored in the same way as in the first case. The porosity was calculated on the basis of the mean specific gravity of nonporous cement and the specific gravity of the experimental specimens.

Further, the influence of delayed cavity filling upon the porosity was investigated. The cavities were represented by small glass tubes, 4 mm in diameter and 2 mm high, and filling of the cavities started between 5 seconds and 3 minutes after completion of mechanical mixing under vacuum. Before measuring the specific gravity the fillings were stored 24 hours at 37°C in 100 % humidity in C-clamps with their end surfaces covered with tinfoil and glass plate.

5. *The compressive strength* was measured using cement mixed by hand or mixed mechanically with or without vacuum. The cylindrical specimens were 3 mm in diameter and 6 mm high. Three minutes after the start of mixing the filled test specimen molds were placed in C-clamps with their end surfaces covered with tinfoil and glass plate in an atmosphere of 100 % humidity and at a temperature of 37°C. One hour later the specimens were withdrawn from the molds and immersed in water at 37°C after previous plane and parallel wet-grinding of the end surfaces on carborundum paper No. 600. When the specimens were 24 hours old their compressive strength was measured in a Losenhausen testing machine at a loading rate of 10 kp per second.

6. *The tensile strength* was measured on specimens prepared in the same way as described under point 5. The tensile strength was measured indirectly by means of diametral compression and with a rate of loading of 2 kp per second. The strength values recorded on the machine were converted into tensile strength from the formula

$$T = \frac{P}{\pi \cdot r \cdot l} \text{ kp/mm}^2$$

where P is the measured strength in kp, r the radius of the specimen in mm, and l its length in mm.

7. *The opacity* was determined in accordance with F.D.I. specification No. 5, except that the specimens were 10 mm in diameter instead of 30 mm.

8. *The water solubility* was measured in accordance with F.D.I. specification No. 5 modified, however, as follows: a) the specimens tested were 10 mm in diameter and 1 mm thick, b) the specimens were not placed in the water until 24 hours after mixing and were kept in water 24 hours, and c) each test was run with 10 specimens instead of 2, and they were placed in the water and removed from it one by one as they had attained the age specified.

9. *Solubility* in 0.05 n unbuffered lactic acid. The specimens (diameter 10 mm, thickness 1 mm) were stored at 37°C in 100 % humidity in C-clamps with their end surfaces covered with tinfoil and glass plates; after 24 hours they were placed in water 144 hours, and then in lactic acid 24 hours. The loss of weight in the lactic acid was determined by weighing the specimens. Further details of this technic will be given in a later work.

RESULTS

Figures 1 and 2 show representative ground and polished sections of silicate cement, which was mixed by hand and mixed mechanically under vacuum,

respectively, using the powder/liquid ratio required for standard consistency.

Table I shows the porosity as affected by the mixing method, while Table II illustrates the effect of delayed cavity filling upon the porosity; the results in Table II are presented graphically in Figure 3. The results in the two tables are obtained with cement mixed to standard consistency. Table III shows the t-values for some of the data in Tables I and II.

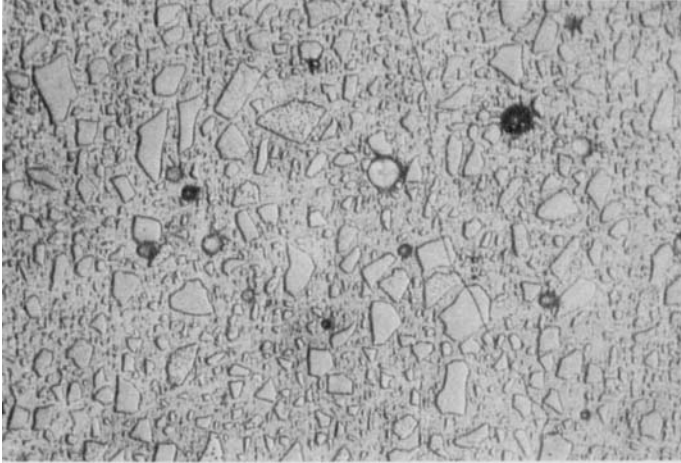


Fig. 1. Porosity in hand-mixed silicate cement.

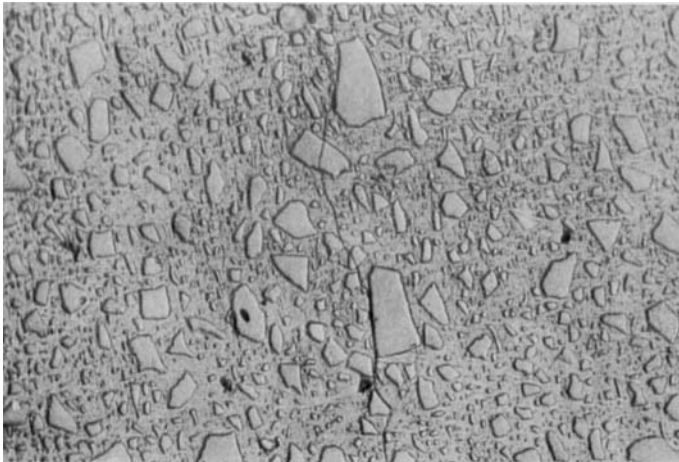


Fig. 2. Porosity in silicate cement mixed mechanically in vacuum.

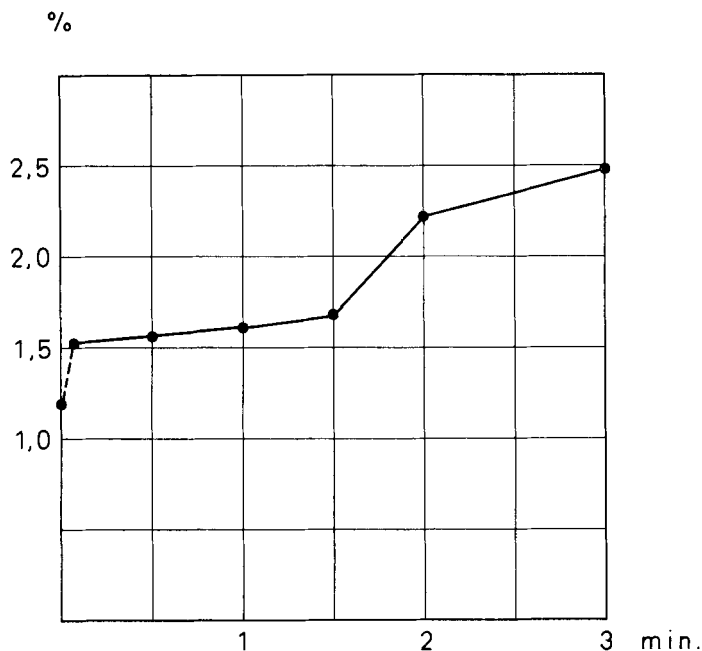


Fig. 3. The effect of delayed cavity filling upon the porosity of silicate cement. The cement was mixed mechanically in vacuum. The values on the abscissa indicate the time from completion of the mix to start of the filling.

Table I.

Influence of the mixing method upon the porosity of silicate cement (%)
n = 5

Hand mixing		\bar{x}	3.35
		a S.D.	0.64
Mechanical mixing		\bar{x}	2.72
		b S.D.	0.20
with vacuum		\bar{x}	1.20
		c S.D.	0.32

The influence of mixing method and powder/liquid ratio upon the compressive strength and the tensile strength of the cement is shown in Tables IV and V, respectively. The proportion 200 mg powder to 0.057 ml liquid represents standard consistency. Figures 4 and 5 illustrate the results graphically, while the graph in Figure 6 shows the correlation between compressive strength and tensile strength.

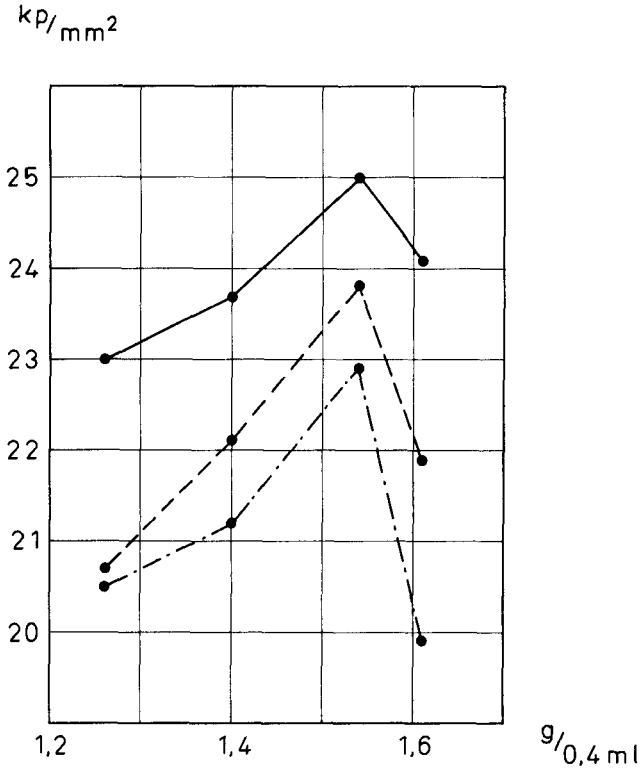


Fig. 4. The compressive strength of silicate cement as affected by powder/liquid ratio and mixing method. The proportion for standard consistency of the cement is 1.4:0.4. Upper curve: mechanical mixing in vacuum. Middle curve: mechanical mixing without vacuum. Lower curve: hand-mixing.

Table II.

The influence of delayed cavity filling upon the porosity of silicate cement fillings (%).

The times indicate the intervals between finishing the mix (mechanical mixing in vacuum) and the beginning of the filling.

n = 10

d		e		f		g		h		i	
5. sec.		$\frac{1}{2}$ min.		1 min.		$1\frac{1}{2}$ min.		2 min.		3 min.	
\bar{x}	S.D.	\bar{x}	S.D.	\bar{x}	S.D.	\bar{x}	S.D.	\bar{x}	S.D.	\bar{x}	S.D.
1.52	0.27	1.57	0.28	1.61	0.28	1.68	0.38	2.22	0.66	2.49	0.36

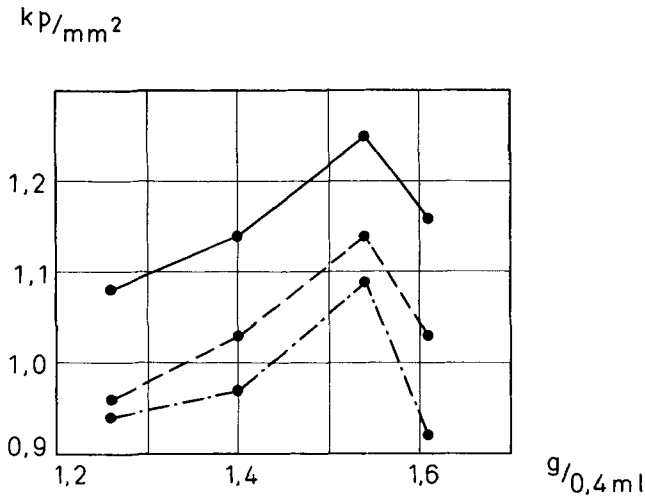


Fig. 5. The tensile strength of silicate cement as affected by powder/liquid ratio and mixing method. Otherwise as Fig. 4.

Table III.
t-values for some of the results in Tables I and II.

a/b	a/c	b/c	c/d	c/e	c/f	c/g	c/h	c/i
	***	***		*	*	*	**	***
2.101	6.700	9.055	1.913	2.210	2.440	2.535	4.037	7.007

* Probably significant. ** Significant. *** Highly significant.

Table IV.
The compressive strength of silicate cement (kp/mm²) as affected by mixing method and powder/liquid ratio.

n = 10

mg powder/0.057 ml liquid	180		200		220		230			
	\bar{x}	S.D.	\bar{x}	S.D.	\bar{x}	S.D.	\bar{x}	S.D.		
Hand mixing	20.5	1.02	21.2	1.11	22.9	1.44	19.9	1.95		
Mechanical mixing	without vacuum		20.7	1.49	22.1	1.33	23.8	1.77	21.9	1.51
	with vacuum		23.0	1.92	23.7	1.77	25.0	2.11	24.1	1.70

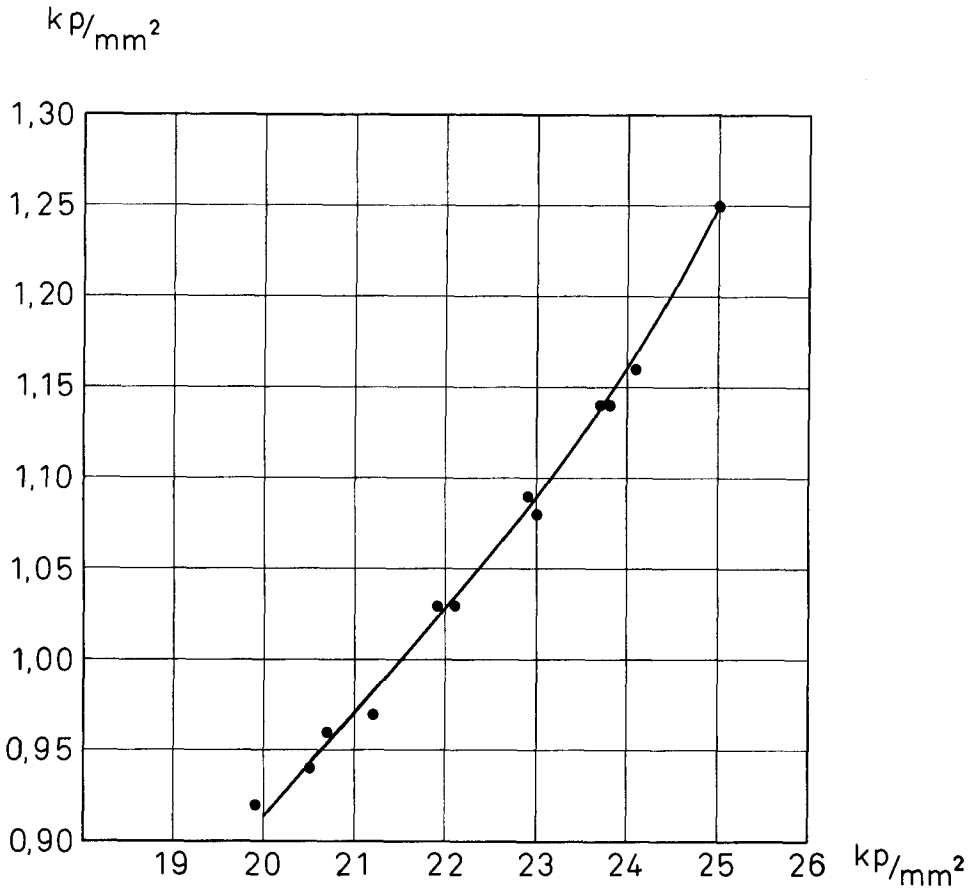


Fig. 6. Correlation between the compressive strength of the cement (abscissa) and its tensile strength (ordinate).

Table V.

The tensile strength of silicate cement (kp/mm²) as affected by mixing method and powder/liquid ratio.

n = 10

mg powder/0.057 ml liquid	180		200		220		230		
	\bar{x}	S.D.	\bar{x}	S.D.	\bar{x}	S.D.	\bar{x}	S.D.	
Hand mixing	0.94	0.11	0.97	0.13	1.09	0.10	0.92	0.13	
Mechanical mixing	without vacuum	0.96	0.09	1.03	0.14	1.14	0.06	1.03	0.10
	with vacuum	1.08	0.14	1.14	0.12	1.25	0.14	1.16	0.16

Examination of the opacity revealed no difference between silicate cement mixed by hand, or mixed mechanically under vacuum.

The results of the solubility tests in water and in 0.05 n lactic acid appear in Table VI. The figures shown for the water solubility tests are mean values for 5 individual measurements, each representing 10 specimens, while the figures for the acid solubility tests are mean values for 10 measurements, each representing one specimen. The solubility is calculated in per cent of the original weight of the specimens.

Table IV.
The silicate cement solubility (%) in water and in 0.05 n lactic acid.

Solvent	Water		0.05 n lactic acid	
	\bar{x}	S.D.	\bar{x}	S.D.
Hand mixing	0.23	0.022	43.6	1.8
Mechanical mixing in vacuum	0.24	0.026	44.1	1.5

DISCUSSION

The reason why the mixing method A described on page 454 proved less effective than method B in reducing the porosity of the cement may be illustrated by the following experiment. A glass tube, open at both ends, with a diameter of 4 mm and a length of 3 mm, was filled with freshly prepared, hand mixed silicate cement and at once placed in a vacuum chamber, where the pressure was brought down to 2 Torr in 5 seconds. As the pressure dropped, a marked increase in the volume of the cement could be observed, presumably owing to the expansion of the air bubbles and, in a less degree, to formation of water vapour bubbles. After re-establishment of normal atmospheric pressure (about 1 minute after the mix was made) the volume of the cement was slightly reduced, though not by far to its original magnitude. Thus the air bubbles in the cement will expand rather easily under vacuum, but have little tendency to collapse under pressure. The explanation is in all probability that the powder particles around the air bubbles are packed together, and act in the same way as the stones in a vault under the influence of the external pressure. A similar »vault effect» is known from many other concentrated mixtures of a powder and a liquid.

To avoid the vault effect the air must therefore be removed from the powder before addition of the liquid (method B, page 454).

The present investigation shows that vacuum-mixing of silicate cement results in a considerable reduction of its porosity; compared with hand-spatulated cement the porosity in the vacuum-mixed cement is only ca. one third.

Filling of a cavity where the cement is placed in increments will result in entrapment of air bubbles between the irregular surfaces of the individual increments, and hence in greater porosity of the filling. Delayed filling adds to the danger of porosity increase (Table II), probably because the surface irregularities increase with the viscosity of the cement.

Of all the properties considered (compressive and tensile strength, opacity, solubility in water and lactic acid) only the strength was altered by the vacuum-mixing. Compared with hand-spatulated cement, the compressive strength increases approximately 10 % and the tensile strength approximately 15 %. Part of the effect found for the vacuum-mixing is, however, due to the incident mechanical mixing; a comparison of the strength of cement mixed mechanically without vacuum and with vacuum shows that the true effect of vacuum is an increase in compressive strength barely exceeding 5 %, and an increase in tensile strength of about 10 %.

The mechanical mixing *per se* reduces the setting time of the cement, and improves its strength properties; the changes are moderate, however.

When comparing these experimental results with those reported by *Lyon & Cosca*, it is in particular noticeable that the two authors were able to demonstrate a reduction in solubility of the cement in both water and acid as a result of the vacuum-mixing (as compared with hand spatulation), while no such effect could be demonstrated in the present study. This discrepancy may be ascribed to the fact that *Lyon & Cosca* measured the solubility in the period from 1—24 hours following mixing of the cement, i.e. before the cement had become stabilized chemically. Thereby the heat produced by mechanical mixing may have influenced the results since it accelerates the setting of the cement. In attempt to avoid an error from this source the solubility tests in the present work were made at a later time when the cement chemically had become more stabilized.

Upon the whole, the effect of mixing silicate in vacuum seems to be rather moderate and of doubtful value in practice.

SUMMARY

The porosity of silicate cement mixed by hand is ca. 3.5 %. It is possible to reduce this porosity to about one third when the mixing is done mechani-

cally in vacuum. One condition for obtaining maximum effect of vacuum-mixing is that the air is eliminated from the powder before addition of the liquid. The porosity of the cement increases by delayed cavity filling. Vacuum-mixing increases the strength of the cement moderately, while other properties (setting time, opacity, and solubility) remain unaffected. It seems doubtful whether vacuum-mixing of silicate cement will result in an improvement of the quality of fillings made of this material.

RÉSUMÉ

MÉLANGE SOUS VIDE DU CIMENT AU SILICATE

La porosité du ciment au silicate malaxé manuellement est d'environ 3,5 %. On peut réduire la porosité à environ un tiers de cette valeur si le mélange est fait mécaniquement et sous vide. Pour que le traitement sous vide soit efficace, il est indispensable que l'air soit éliminé de la poudre avant l'addition du liquide. La porosité du ciment augmente lorsque l'obturation de la cavité est différée. Le malaxage sous vide détermine une augmentation modérée de la résistance mécanique du ciment, mais ne semble pas influencer sur d'autres propriétés (temps de prise, opacité et solubilité). Les auteurs doutent que le malaxage sous vide du ciment au silicate puisse déterminer une amélioration notable de la qualité des obturations faites avec ce matériau.

ZUSAMMENFASSUNG

VAKUUMMISCHUNG VON SILIKATZEMENT

Die Porosität in von Hand ausgerührtem Silikatzement beträgt ca. 3,5 %. Es ist möglich, diese Porosität auf etwa ein Drittel zu reduzieren, wenn die Mischung mechanisch und unter Vakuum geschieht. Die Voraussetzung dafür, dass die Vakuumbehandlung effektiv sein kann, ist, dass die Luft aus dem Pulver eliminiert ist, ehe die Flüssigkeit beigemischt wird. Die Porosität des Zements steigt bei retardierter Kavitätenfüllung. Die Vakuummischung bewirkt eine moderate Erhöhung der Festigkeit des Zements, während andere Eigenschaften (Abbindungszeit, Opazität und Lösbarkeit) unbeeinflusst erscheinen. Es muss bezweifelt werden, dass die Vakuummischung von Silikatzement eine Qualitätsverbesserung von Füllungen aus diesem Material bewirkt.

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