

# Impact of water quality on setting of irreversible hydrocolloid impression materials

Pavel Bradna, PhD,<sup>a</sup> and Darina Cerna, MSc<sup>b</sup>

The Institute of Dental Research, First Medical Faculty of Charles University and General University Hospital, Prague, Czech Republic; Spofa-Dental a. s., Prague, Czech Republic

**Statement of problem.** Setting of irreversible hydrocolloid impression materials is based on the ionic reaction between carboxylic groups and calcium ions and may, therefore, be affected by ionic species present in the mixing water. The impact of this phenomenon on the clinical performance of these materials has not been well documented.

**Purpose.** The purpose of this study was to compare the setting behavior of irreversible hydrocolloid impression materials when mixed with tap and distilled water, and to determine the impact of typical cations present in tap water and their concentrations on the setting process.

**Material and methods.** Six brands of irreversible hydrocolloid impression materials (Kromopan 100, Xantalgin Select FS, Alginoplast, Elastic Plus, Ypeen, and Ypeen Premium) were mixed with tap and distilled water (control) according to manufacturers' recommendations. Elastic Plus was also mixed with aqueous solutions containing various concentrations of NaCl, CaCl<sub>2</sub>, and AlCl<sub>3</sub> to determine the role of typical cations on setting. Using a controlled shear stress oscillatory rheometer, time changes of storage ( $G'$ ) and loss ( $G''$ ) moduli during setting were measured at 23°C and used to determine the working and setting times and rigidity of set impression materials. The sample size ( $n=3$ ) for each material/mixing system was increased to 8 to increase reliability of measurements in systems where the effect of mixing water was low or variance of results was high. The data were analyzed ( $\alpha=.05$ ) using a  $t$  test (tap water), a 1-way ANOVA, a Tukey post hoc test (shear stress), and a nested ANOVA and Fisher Least Significant Difference post hoc analysis (cation and cation concentration).

**Results.** Statistical analysis showed significant ( $P<.001$ ) acceleration in the setting rate for Kromopan 100, the working time of which was shorter with tap water by 23.4 seconds and the setting time, by 32.8 seconds. Similar significant reductions (in seconds) in both working and setting times, respectively, were found with Xantalgin Select FS (12.0,  $P<.001$  and 23.6,  $P<.002$ ), Alginoplast (24.3,  $P<.001$  and 44.7,  $P<.005$ ), and Elastic Plus (23.0,  $P<.001$  and 22.0,  $P<.002$ ), when compared with the same materials mixed with distilled water. At the same time, the rigidity of the set impression materials mixed with tap water increased by approximately 10%. With Ypeen Premium, the setting time decreased significantly by 20.0 seconds ( $P<.001$ ) with tap water, while the working time and rigidity did not change. The setting behavior of Ypeen was not significantly affected by water quality. Acceleration of the setting reactions and increase in impression rigidity depended ( $P<.001$ ) primarily on concentration and valency of cations present in mixing water.

**Conclusion.** Setting of irreversible hydrocolloid impression materials can be significantly accelerated when tap water with high water hardness is used for mixing or when the mixing water contains higher concentrations of cations such as Na<sup>+</sup>, Ca<sup>2+</sup>, and Al<sup>3+</sup>. (J Prosthet Dent 2006;96:443-8.)

## CLINICAL IMPLICATIONS

*This study suggests that to gain optimal properties of irreversible hydrocolloid impression materials, distilled or demineralized water should be used for mixing these materials in regions where tap water is hard.*

Dental irreversible hydrocolloid impression materials rank among the most frequently used impression materials in dentistry.<sup>1</sup> They are easy to prepare and

manipulate, comfortable for patients, fast setting, and low in cost.<sup>1</sup> Irreversible hydrocolloids are produced as powders containing soluble alginate salt, calcium sulfate (hemihydrate or dihydrate), and fillers as primary components.<sup>1-3</sup> After mixing with water, carboxylate groups attached to alginate chains react with calcium ions, producing an insoluble calcium salt, which is accompanied by gelling of the material. To control the working and setting times and to adjust mechanical properties of

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<sup>a</sup>Research Scientist, The Institute of Dental Research, First Medical Faculty of Charles University and General University Hospital.

<sup>b</sup>Research Scientist, Research and Development Department, Spofa-Dental a. s.

the impression materials, various ingredients are added, including retarders (usually sodium phosphates), and the material is tested using distilled or demineralized water as required in the International Organization for Standardization (ISO) standard.<sup>4</sup>

In clinical practice, however, tap water is generally used to mix these impression materials rather than distilled or demineralized water. Tap water usually contains high and unpredictable levels of metal ions,<sup>5</sup> primarily calcium and magnesium. A measure of their concentration is water hardness, expressed in milligrams per liter (mg/L) of calcium and magnesium carbonates dissolved in water. Because these cations contained in mixing water can also react with negatively charged carboxylic groups on alginate chains, the use of tap water may influence the setting of irreversible hydrocolloid materials and cause shifts in their properties from those stated by the manufacturer. Despite the known influences that constituent ions in tap water may have on the clinical performance of irreversible hydrocolloids, this phenomenon has not been adequately investigated.

During the setting process, a 3-dimensional network of alginate chains cross-linked with calcium ions is formed, which results in a gradual change of viscous to elastic properties of the mixed impression materials. Since rheological methods can accurately reflect these changes, they are suitable to characterize the working and setting times and rigidity of impression materials.<sup>6-10</sup>

The purpose of this study was to investigate the effect of tap water rather than distilled water on the working and setting times and rigidity of 6 irreversible hydrocolloid impression materials, and secondly, to determine the effect of various concentrations of Na<sup>+</sup>, Ca<sup>2+</sup>, and Al<sup>3+</sup>, as typical cations usually present in tap water, on these characteristics. The null hypothesis was that there is no impact of water quality on the working and setting times of irreversible hydrocolloid impression materials.

## MATERIAL AND METHODS

The 6 irreversible hydrocolloid impression materials used in this study are shown in Table I. All powders were hand mixed with tap and distilled (control) water at a powder/water ratio recommended by the manufacturer (Table I). Elastic Plus was also mixed with aqueous solutions containing various concentrations of NaCl, CaCl<sub>2</sub>, and AlCl<sub>3</sub> to determine the role of typical cations on its setting. The powders, water, mixing bowl, and spatula were stored at 23°C ± 1°C and 50% ± 10% relative humidity for at least 10 hours before testing. Tap water from Prague City (Czech Republic) was used for mixing. The concentrations of Na<sup>+</sup>, Ca<sup>2+</sup>, Mg<sup>2+</sup>, Al<sup>3+</sup>, and Fe<sup>3+</sup> ions contained in the water

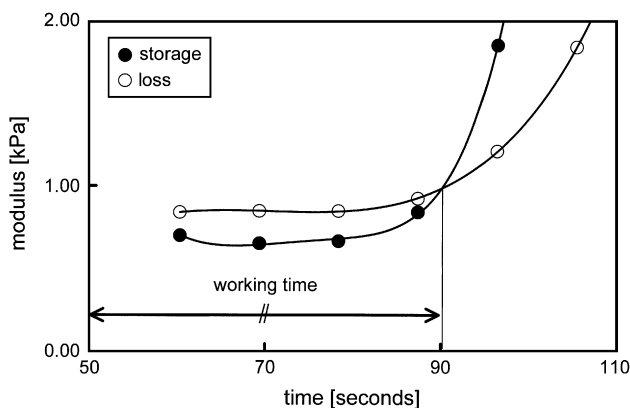
**Table I.** Irreversible hydrocolloid impression materials used in this study

Product	Manufacturer	Powder/water (g/mL)	Batch no.
Kromopan 100	Lascod, S.p.A., Florence, Italy	9/20	014630
Xantalgin Select FS	Bayer, Leverkusen, Germany	9/21	0358710
Alginoplast	Bayer	9/21	0358733
Elastic Plus	Spofa-Dental a.s., Prague, Czech Rep	9/20	166466
Ypeen	Spofa-Dental a.s.	10/20	106849
Ypeen Premium	Spofa-Dental a.s.	9/20	741227

FS, Fast setting.

were determined by atomic absorption spectroscopy as amounting to 16 mg/L, 95 mg/L, 6.8 mg/L, 0.02 mg/L, and 0.14 mg/L, respectively. Also, the anions Cl<sup>-</sup>, SO<sub>4</sub><sup>2-</sup>, and NO<sub>3</sub><sup>-</sup> and very small concentrations of NO<sub>2</sub><sup>-</sup> anions were contained in the water, not exceeding a total concentration of 100 mg/L. To prepare solutions of the required salt concentration, analytical grade NaCl, CaCl<sub>2</sub>, and AlCl<sub>3</sub> salts (Lachema a.s., Neratovice, Czech Republic) were dissolved in distilled water.

Working and setting times and rigidity of set impression materials were measured using a controlled shear stress oscillatory rheometer (Rotovisco RT 10; Haake, Karlsruhe, Germany). In oscillatory tests,<sup>11</sup> a material is subjected to a small shear stress that varies sinusoidally with time. In this experiment, mixed paste was transferred between 2 parallel circular plates of the rheometer. When a measurement began, the upper plate was forced to move harmonically at a given frequency, back and forth, with the shear stress changing from zero to a predetermined maximum amplitude. The material deformation (strain) in response to the shear stress and the angle between stress and strain response, called the phase lag,  $\delta$ , were determined during the test.<sup>9-10</sup> If the material is an ideal elastic solid and behaves like a spring, then  $\delta$  equals 0. For viscoelastic materials, such as hydrocolloid impression materials, which are neither ideal fluids nor ideal elastic solids, the phase lag changes from approximately 70 degrees, corresponding to fluid (viscous) behavior of mixed material, to 5 degrees for the set material.<sup>6</sup> If the stress amplitude is sufficiently small, the material behaves in a linear viscoelastic manner—that is, its properties do not depend on the shear stress imposed. In this situation, the shear storage modulus  $G'$  and shear loss modulus  $G''$  characterizing elastic and viscous properties of setting material can be calculated from stress and strain amplitudes and the phase lag using the following equations<sup>11</sup>:



**Fig. 1.** Typical changes of storage  $G'$  and loss  $G''$  loss moduli in pre-gel phase of setting curve and definition of working time.

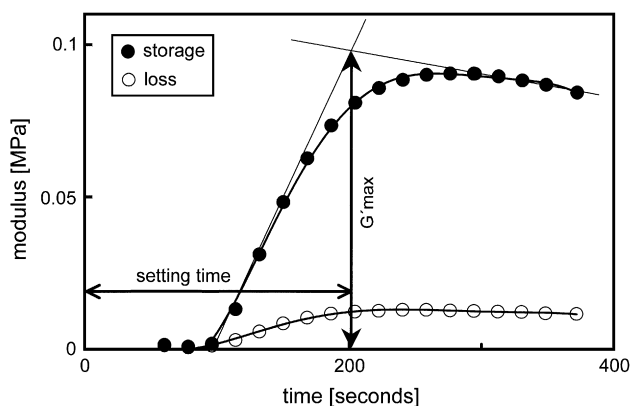
$$G' = \tau_0 \cos \delta / \gamma_0 \quad (1)$$

$$G'' = \tau_0 \sin \delta / \gamma_0 \quad (2)$$

where  $\tau_0$  and  $\gamma_0$  are the stress and strain amplitudes, respectively. In measurements of viscoelastic properties, both moduli are usually recorded as a function of angular frequency. However, in experiments focused on time-dependant changes in viscoelastic properties, a measurement at a constant angular frequency is performed, which provides a convenient method to characterize the setting of impression materials.<sup>6,9-10</sup>

Before testing, it was necessary to determine the shear stress at which the properties of setting materials remained in the linear viscoelastic region. For this reason, Elastic Plus, as a representative of the irreversible hydrocolloid impression materials under study, was tested at the shear stress of 100, 300, and 500 Pa and at the angular frequency 2.15 Hz used throughout the study. All the measurements were performed at  $23^\circ\text{C} \pm 0.1^\circ\text{C}$  using a stainless steel parallel-plate geometry with a serrated surface to avoid slippage of the material. The separation distance between the plates was adjusted to approximately 1.5 to 2.0 mm. After being mixed according to the manufacturer's instruction, approximately 2 mL of paste were immediately transferred between the plates. Excessive impression material was removed after the distance between the plates was adjusted. The first data were recorded approximately 60 seconds after the start of mixing. The mixing time was 30 seconds for all materials tested. All moduli calculations were performed using the software provided with the rheometer.

When the measurement started, data of shear stress, shear strain, and phase lag were automatically recorded in 10-second intervals. After the first 5 minutes, the data recording interval was switched to 20 seconds. The run was finished 45 minutes after the start of mixing. To determine the working and setting times of the impression materials,  $G'$  and  $G''$  versus time were



**Fig. 2.** Full-range setting curve and definitions of setting time and maximum storage modulus  $G'_{\max}$ .

**Table II.** Working ( $t_w$ ) and setting ( $t_s$ ) times and maximum storage modulus  $G'_{\max}$  (mean  $\pm$  SD,  $n=3$ ) of Elastic Plus when mixed with distilled water measured at different maximum shear stress values

Shear stress (Pa)	$t_w$ (s)	$t_s$ (s)	$G'_{\max}$ (MPa)
100	105.4 $\pm$ 1.0 <sup>b</sup>	210.6 $\pm$ 3.0 <sup>a</sup>	0.13 $\pm$ 0.02
300	110.0 $\pm$ 2.0 <sup>a</sup>	218.0 $\pm$ 5.5 <sup>a</sup>	0.08 $\pm$ 0.01 <sup>a</sup>
500	108.6 $\pm$ 2.2 <sup>a,b</sup>	216.2 $\pm$ 5.3 <sup>a</sup>	0.08 $\pm$ 0.01 <sup>a</sup>

Identical superscripted letters in columns indicate no significant difference ( $P > .05$ ).

**Table III.** ANOVA of effect of shear stress on working time, setting time, and maximum storage modulus

Effect	Sum of squares	df	Mean square	F	P
Working time					
Intercept	174970	1	174970	43462	<.001
Shear stress	57.6	2	28.8	7.2	<.001
Error	48.3	2	4.0		
Setting time					
Intercept	692945	1	692945	27717	<.001
Shear stress	148.9	2	74.5	3.0	.09
Error	300	12	25.0		
Maximum storage modulus					
Intercept	14.6	1	14.6	909	<.001
Shear stress	48.9	2	0.35	22.0	<.001
Error	0.19	12	0.016		

recorded. The working time was defined as the time interval from the start of mixing to the crossover of the  $G'$  and  $G''$  (Fig. 1), which corresponded to  $\delta=45$  degrees.<sup>6</sup> The setting time was defined as the time interval from the start of mixing to the intercept of the tangent drawn at the inflection point of  $G'$  versus time dependence and a tangent drawn in the region where the material was set (Fig. 2). The maximum storage

**Table IV.** Working ( $t_w$ ), setting ( $t_s$ ) times (mean  $\pm$  SD) and respective differences ( $\Delta$ ) when mixed with distilled (D) and tap (T) water

Product	Medium	n	$t_w$ (s)	$\Delta$ (s)	$t_s$ (s)	$\Delta$ (s)	Significance $P(t_w)/P(t_s)$
Kromopan 100	D	3	122.9 $\pm$ 3.5	23.4	258.1 $\pm$ 3.2	32.8	<.001/<.001
	T	3	99.5 $\pm$ 1.6		225.3 $\pm$ 3.0		
Xantalgin Select FS	D	5	105.3 $\pm$ 3.2	12.0	192.9 $\pm$ 7.0	23.6	<.001/<.002
	T	4	93.3 $\pm$ 1.2		169.3 $\pm$ 10.5		
Alginoplast	D	3	164.6 $\pm$ 2.4	24.3	315.0 $\pm$ 6.8	44.7	<.001/<.005
	T	3	140.3 $\pm$ 1.6		270.3 $\pm$ 1.2		
Elastic Plus	D	5	110.0 $\pm$ 2.0	23.0	218.0 $\pm$ 5.5	22.0	<.001/<.002
	T	5	87.0 $\pm$ 4.0		196.0 $\pm$ 9.0		
Ypeen	D	4	75.7 $\pm$ 5.5 <sup>a</sup>	-6.3	187.5 $\pm$ 1.6 <sup>a</sup>	-10.6	.27/.082
	T	4	82.0 $\pm$ 8.9 <sup>a</sup>		198.1 $\pm$ 10.0 <sup>a</sup>		
Ypeen Premium	D	6	92.0 $\pm$ 2.0 <sup>a</sup>	1.4	247.8 $\pm$ 8.8	20.0	.11/<.001
	T	8	90.6 $\pm$ 1.0 <sup>a</sup>		227.8 $\pm$ 7.7		

Identical superscripted letters indicate no significant difference ( $P>.05$ ); n indicates number of measurements. FS, Fast setting.

**Table V.** Results of nested ANOVA for working time

Effect	Sum of squares	df	Mean square	F	P
Intercept	241974	1	41974	14060	<.001
Cation	1978	2	989	57.5	<.001
Concentration (cation)	4875	8	609	35.4	<.001
Error	47.5	26	17		

**Table VI.** Results of nested ANOVA for setting time

Effect	Sum of squares	df	Mean square	F	P
Intercept	1442224	1	1442224	38462	<.001
Cation	6744	2	3372	89.9	<.001
Concentration (cation)	20884	10	2088	55.7	<.001
Error	1275	34	37		

modulus  $G'_{max}$  was determined from the intercept of tangents used to determine the setting time (Fig. 2). The initial sample size was 3. For the impression material/mixing systems where the effect of water or cation concentration was low or the variance of data was higher, the sample size was increased to 8 to improve the reliability of the results. The mean values obtained in tap water and distilled water (control) were analyzed using  $t$  tests for not-equal variances. A 1-way analysis (ANOVA) and a Tukey post hoc test were used to compare data ( $n=3$ ) obtained at various shear stresses, and a nested ANOVA with a Fisher post hoc test ( $n=3-8$ ), to analyze the effect of various cations and their concentrations. Calculations ( $\alpha=.05$ ) were performed using statistical software (Statistica 6; StatSoft Inc, Tulsa, Okla).

## RESULTS

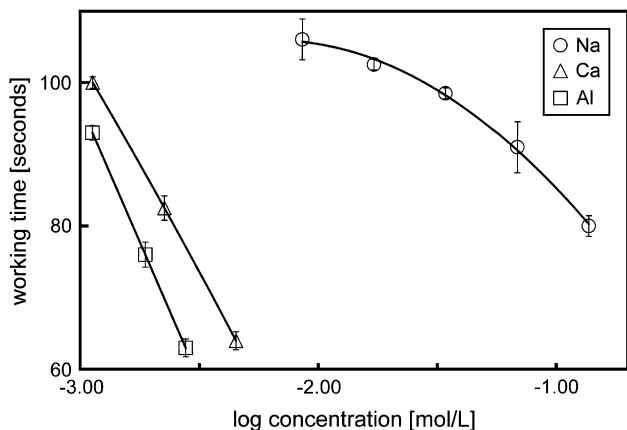
The mean values of the working and setting times and  $G'_{max}$  (Table II) measured at different levels of maximum shear stress and analyzed with a 1-way ANOVA (Table III) revealed that at the maximum shear stress of 300 and 500 Pa these characteristics were not statistically different. For this reason, a maximum shear stress of 300 Pa was used in this study. The summarized data (Table IV) show that working and setting times for Kromopan 100, Xantalgin Select FS, Alginoplast, and

Elastic Plus were significantly shorter when tap water was used instead of distilled water. Also, higher values of  $G'_{max}$ , which increased by approximately 10%, indicated an increase in rigidity of the set impression materials. With Ypeen Premium, only the setting time was significantly shorter when tap water was used, and for Ypeen, no significant effect of water quality on its setting was observed (Table IV).

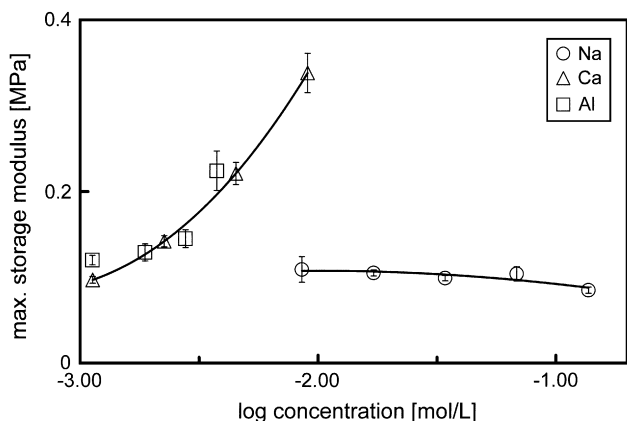
Experiments with increasing concentrations of  $Na^+$ ,  $Ca^{2+}$ , and  $Al^{3+}$  in the mixing water, with Elastic Plus as a representative of impression materials sensitive to water quality, and compared using a nested ANOVA demonstrated ( $P<.001$ ) (Tables V and VI) that rising cation valency and cation concentration significantly accelerated set of the material (Figs. 3 and 4). Multivalent cations also influenced  $G'_{max}$  (Fig. 5), which increased significantly ( $P<.001$ ) (Table VII) with concentrations of  $Ca^{2+}$  and  $Al^{3+}$  cations, reflecting higher rigidity of set materials due to an increasing number of crosslinks formed between alginate chains during setting.

## DISCUSSION

In clinical practice, the working and setting times of impression materials are important characteristics. To obtain the optimum accuracy of an impression and

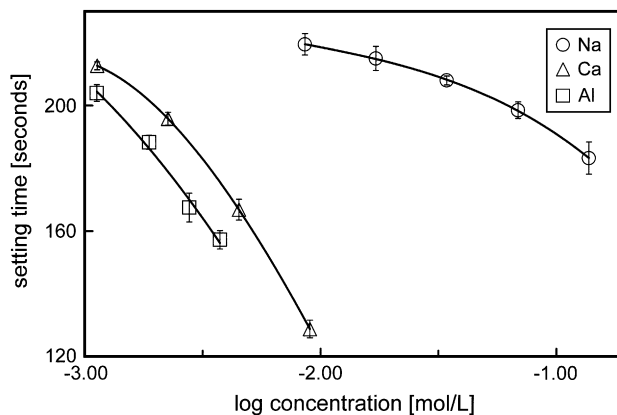


**Fig. 3.** Dependence of working time of Elastic Plus on concentration of various cations.



**Fig. 5.** Dependence of maximum storage modulus of Elastic Plus on concentration of various cations.

make the impression-making procedure convenient for both dentist and patient, the manufacturer determines the values of these characteristics. The ISO standard<sup>4</sup> defines the total working time as a period between the start of mixing and the commencement of setting, measured by an indentation test. The setting time is likewise defined as a period of time between the start of mixing and the achievement of the necessary elasticity to remove an impression. Because of experimental difficulties, measurement of setting time is not required. Instead, this characteristic is evaluated by the testing recovery from deformation, the value of which should amount to at least 95% at the setting time stated by the manufacturer.<sup>4</sup> As the indentation test reflects viscous rather than elastic properties, some attempts to determine the working and setting times of impression materials using rheological methods have been published.<sup>6-10</sup> However, due to a lack of a relationship between rheological characteristics and clinically significant parameters such as working and setting times, a time interval at which the phase lag reached 45 or 60



**Fig. 4.** Dependence of setting time of Elastic Plus on concentration of various cations.

**Table VII.** Results of nested ANOVA for maximum storage modulus

Effect	Sum of squares	df	Mean square	F	P
Intercept	9178	1	9178	1648	<.001
Cation	660	2	330	59.5	<.001
Concentration (cation)	1323	10	132	23.8	<.001
Error	178	32	5.6		

degrees was described as a measure of the working time. Similar to Shigeto et al,<sup>6</sup> a phase lag equal to 45 degrees was used in this study for determination of the working time, in that a transformation of a system from a viscous to a solid elastic body occurs due to crosslinking reactions. The setting time was derived from the time interval corresponding with the point at which a steep increase of the elastic modulus changed into a steady-state value, typical for the final stage of setting (Fig. 2).

The primary objective of this study was to evaluate the impact of mixing water quality on both working and setting times and on the rigidity of typical irreversible hydrocolloid impression materials. It is clear from the results in Table IV that when other variables are controlled, the quality of mixing water may cause significant changes in the setting process. Thus, the null hypothesis that the setting of irreversible hydrocolloid impression materials does not depend on the quality of mixing water was rejected, except for Ypeen. The differences between the working and setting times stated in the manufacturers' instructions and the actual behavior of the materials may negatively affect the accuracy of an impression. It is known that the setting of irreversible hydrocolloid impression materials depends not only on composition, but may be affected by other variables such as mixing ratio, temperature, and storage conditions.<sup>1</sup> When the typical working time of irreversible

hydrocolloid impression materials is approximately 90 seconds, including 30 to 45 seconds for mixing, a reduction by 12 to 24 seconds significantly shortens the manipulation time for these materials.

A comparison of the effects of univalent  $\text{Na}^+$  and multivalent  $\text{Ca}^{2+}$  and  $\text{Al}^{3+}$  used as model species (Figs. 3 through 5) suggests that it is the presence of divalent and trivalent cations that are responsible for this behavior. Mixing an irreversible hydrocolloid impression material with water begins various physicochemical processes,<sup>1</sup> including dissolution of calcium sulfate and release of free  $\text{Ca}^{2+}$  ions, dissolution of a retarder, swelling and dissolution of alginate particles, reaction between  $\text{Ca}^{2+}$  and anions from the retarders resulting in insoluble Ca salts, and reaction of alginate carboxylate groups with  $\text{Ca}^{2+}$  ions leading to setting of the material. With reference to this reaction scheme, the effect of multivalent cations present in tap water might be interpreted as follows.  $\text{Ca}^{2+}$  and other multivalent ions present in the mixing water may react immediately with soluble anions of the retarder, decreasing its effective concentration adjusted to delay the setting of the impression material. As a result of the lower retarder concentration, a reduction in the working and setting times may be observed. Due to the excess of  $\text{Ca}^{2+}$  ions available to the reaction with carboxylic groups on alginate chains, the crosslink density and, thus, rigidity of the set materials increase, as demonstrated by higher  $G'_{\text{max}}$  values. Such behavior depends on individual compositions of various brands of irreversible hydrocolloid impression materials. Currently, many of these materials are produced with a color indication of various gelation stages to simplify their manipulation. Further research should be focused on these materials. Such investigation, however, should be performed not only at 23°C, which was a limitation of this study, but also at higher temperatures to mimic setting under intraoral conditions.

## CONCLUSIONS

Within the limitations of the study, when tap water with higher water hardness relative to distilled water is

used to mix the 6 irreversible hydrocolloid materials tested, shortening of working and setting times and an increase in material rigidity was demonstrated. This effect depends, however, on the water hardness and is product specific.

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### Reprint requests to:

DR PAVEL BRADNA  
THE INSTITUTE OF DENTAL RESEARCH FIRST MEDICAL FACULTY OF  
CHARLES UNIVERSITY AND GENERAL HOSPITAL  
PRAGUE 2, VINOHRADSKA 48  
CZECH REPUBLIC 120 00  
FAX: 00420 224 247 034  
E-MAIL: [bradna@vus.cz](mailto:bradna@vus.cz)

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