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Self Disinfecting Reversible Hydrocolloid Impression Gels: Effect of Composition and Nanosilver on Characteristic Properties and Gelation Temperature

S. Azemat Miri[1], G. Mir Mohamad Sadeghi[2,*] and M. Rabiee[1]

[1]Department of Biomedical Engineering, Amirkabir University of Technology; [2]Department of Polymer Engineering and Color Technology, Iran

Abstract: We have developed a new hydrocolloid impression gel to eliminate disinfection process that caused to inaccuracy in dimensions of final mold. At first, the proper sample has been prepared based on recommended formulation in literature as well as trial and errors *via* experimental works. Formulations prepared by variation amounts of ingredients and also samples prepared by adding nanosilver as an antibacterial agent. Characteristic properties such as tear strength, gel temperature, compressive strength and elastic recovery of prepared hydrocolloid impression gels have been evaluated. The effect of agar, potassium sulfate and di-sodiumtetraborate (Borax) on characteristic properties has been investigated. In addition, role of nanosilver as an antibacterial which can be eliminate disinfecting process, on final properties of gels has been evaluated this work presents the role of ingredient on properties of self disinfecting agar impression gel.

Keywords: Agar hydrocolloid, impression materials, nanosilver.

INTRODUCTION

Impression materials are used to record intraoral structures for the fabrication of definitive restorations. Accurate impressions are necessary for construction of any dental prosthesis. The accuracy of these final restorations depends greatly on the impression materials and techniques. Impression materials that are currently popular include polyethers, addition silicones, polysulfides, and hydrocolloids. With elastomeric impression materials such as polyvinyl siloxane, polyether, and polysulfide, the dimensional accuracy is usually time dependent, with greater dimensional accuracy occurring immediately after polymerization is complete but declining as the impression is stored for extended periods of time. Hydrocolloids have a high hydrophilic nature that allows this material to capture accurate impressions in the presence of some saliva or blood. It has a low wetting angle so it easily captures full arch impressions. It has moderate ability to reproduce detail and costs relatively little compared with other impression materials. It is not accurate enough for fixed partial dentures but is used for partial framework impressions [1]. Agar is a gel-forming polysaccharide, widely used in industry and in medical applications. It is extracted from the walls of certain red seaweeds or agarophytes. Agar hydrocolloid impression materials are compounded from reversible agar gels. When heated, they liquefy or go into the sol state, and on cooling they return to the gel state. This type of gels known as reversible due to their repeatability, while irreversible gels can't be used again [2].

Reversible hydrocolloid was introduced to the dental profession in 1925 by Alphons Poller, an Austrian as impression material [3]. Agar provides a thermoreversible gel that sol-gel formation is reversible depends on temperature. This material forms a colloid after absorbing water, which liquefies between 71°C and 100°C and sets to a gel again between 30°C and 50°C, depending on concentration of the agar [2]. After preparing the mold by impression gel, it should be disinfected to prevent spread of contamination. It also proved that disinfection process affects on dimensional stability.

In this study, agar and agar-nanosilver hydrocolloids as reversible impression materials have been prepared using a modified formulation and characteristic properties such as tear strength, gel temperature, compressive strength and elastic recovery of the gels have been evaluated. The effect of agar, potassium sulfate and di-sodiumtetraborate (Borax) on mentioned properties have been evaluated. In addition, role of nanosilver as an antibacterial to eliminate disinfecting process on these properties, has been investigated.

EXPERIMENTAL

Materials

High gel strength agar (Sigma A 6924), Potassium sulfate, di-sodiumtetraborate-10 hydrate ($Na_2B_4O_7$, $10H_2O$) and silicon oil were from Merck.

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*Address corresponding to this author at the Department of Polymer Engineering and Color Technology, Amirkabir University of Technology, 15875/4413, Tehran, Iran; Tel: +98-21-64542442; Fax: +98-21-66468243; E-mail: gsadeghi@aut.ac.ir

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Table 1: Composition of Agar Hydrocolloid Prepared Impression

	Agar	Potassium sulfate (g)	Borax (g)	Water (g)	Silicon oil (g)
A1	6.90	0.8	1.41	90.87	0.03
A2	5.70	0.8	1.41	92.07	0.03
B1	6.35	0.8	2.00	90.83	0.03
B2	6.35	0.8	0.80	92.02	0.03
C1	6.35	1.4	1.41	90.82	0.03
C2	6.35	0.2	1.41	92.02	0.03
D	6.35	0.8	1.41	91.42	0.03

Nanosilver solution (4000 ppm) was purchased from Nanonasbe pars, Iran (Nanocid). Before using nanosilver, uv-absorpsion spectrum has been prepared. A peak with maximum at about 416 nm is characteristic for nanosilver [4]. According to other reports, minimum inhibitory concentration (MIC) for antimicrobial effect of Ag-NPs against *S. aureus* and *E. coli* is 5 and 10 ppm, respectively [5] and in β-Chitin/nanosilver scaffold is 1 and 1.56-6.25 µg/ml [6]. Therefore in this work, the concentration of nanosilver was chosen as 500 ppm.

Techniques

Thermo HAAKE DC10 water bath is used to heat gel at set temperature, metallic cylindrical mold and metallic mold for measuring compression and recovery from deformation, Metal tray for measuring of gel point and a specific metal mold for tear testing according to ISO1564 (ANSI/ADA NO.11) [7, 8] are used.

Tear and compression strength tests on gels after setting were carried out at room temperature using a Testometric-M350, 5 KN mechanical testing machine with a crosshead speed of 50 mm/min, capable to operating at tensile and compression mode. A dial indicator was used to measure the recovery from deformation.

Gel Preparation

Used formulations to prepare agar impression are listed in Table **1**. At first, sample D has been prepared based on recommended formulation in literature as well as trial and errors *via* experimental works then; other formulations have been made to understand effect of each component. A1 and A2 are different in amount of agar, as ±0.6g adding or subtracting of agar respect to the sample D. B1 and B2 are different in amount of borax (to increase the strength), as ± 0.6g of borax respect to the sample D. C1 and C2 are different in amount of potassium sulfate (to accelerate the plaster setting), as ±0.6 adding or subtracting respect to the sample D.

To prepare impression gel, agar powder was mixed with water in the beaker at room temperature. Borax and potassium sulfate were dissolved separately in residue of water and gently shacked and added to beaker. Finally, silicon oil was added.

To prepare self-disinfecting gel, sample D which was the best sample, has been chosen. Then according to Table **2**, a nanosilver solution has been added to the beaker before putting it in the bath. Table **2** shows self disinfecting gels containing nanosilver.

The beaker placed in the water bath at 85-88ºC. After 10-15 min the beaker was removed and solution was poured into metallic cylindrical molds for compression and recovery from deformation test and also specified mold for tear strength test. Dimensional of cylindrical molds were 16×20, 18×20 Cm ×Cm.

Then, solutions (sol state of agar compound) have been cooled in the molds at room temperature for 15 min. All tests were carried out at 15min after cooling in the room temperature.

Table 2: Composition of Self Disinfecting-Agar Hydrocolloid Impression

	Agar	Potassium sulfate (g)	Borax (g)	Water (g)	Silicon oil (g)	nanosilver (ml)
D1	6.35	0.80	1.40	78.92	0.030	12.50

Table 3: Gel Point, Tear Strength, Recovery from Deformation, Compressive Strength (According to ISO1564, ANSI/ADA NO.11)

Compressive* strength (MPa)	Recovery from deformation (%)	Tear strength (N/mm)	Gel point ºC	Sample
0.47-0.79	>95	0.80-1.00	44-46	A1
0.22-0.44	95-96	0.45-0.55	36-38	A2
0.37-0.68	92-94	0.50-0.57	44-46	B1
0.20-0.40	93-94	0.65-0.97	34-36	B2
0.59-0.78	94-96	0.55-0.71	44-45	C1
0.37-0.65	93-95	0.73-1.00	35-38	C2
0.70-1.10	>95	0.7-0.90	39-41	D
0.70-0.95	96-98	0.7-0.85	38-41	D1

*Tested using two rates of loading: 254 mm/min and 48 mm/min.

Evaluation of Characteristic Properties

Gel Point of Prepared Hydrocolloid

Sol-state formation of compound poured into specified metal tray while a thermometer placed into a tray through a hole on the body of tray. When a test tube (metallic) impressed to gel and no material has been adhered to the tube surface, the gelation temperature recorded [7-8].

Five tests were carried out on each specimen and mean average values have been reported. To evaluate modulus-temperature behavior, gel-temperature has been tested through rheological method [9] for sample D1. Test results are listed in Table **3**, Figures **1** and **2**.

Tear Strength

A Universal machine (Testometric-M350, 5KN) equipped with two pneumatic grips is used to measure tear strength of samples. The grips were coated with adhesive-backed paper in order to inhibit of skidding and an effective gripping in pressure of about 137,895 Pa. The prepared specimen removed from mold and thickness was measured at a point on the midline nearest the V-notch in millimeter (d) as shown in Figure **3** Then specimen positioned in the grips and maximum tensile force (F) at a cross-head rate of 500mm/min was recorded. Five tests were carried out on each specimen and mean average values have been reported.

Tear strength (Ts) of gel is calculated according to Formula 1:

$$F = Ts \times d \qquad (1)$$

Where, F, is maximum force in Newton

d, is thickness of midline nearest the V-notch in millimeter.

Ts, Tear strength

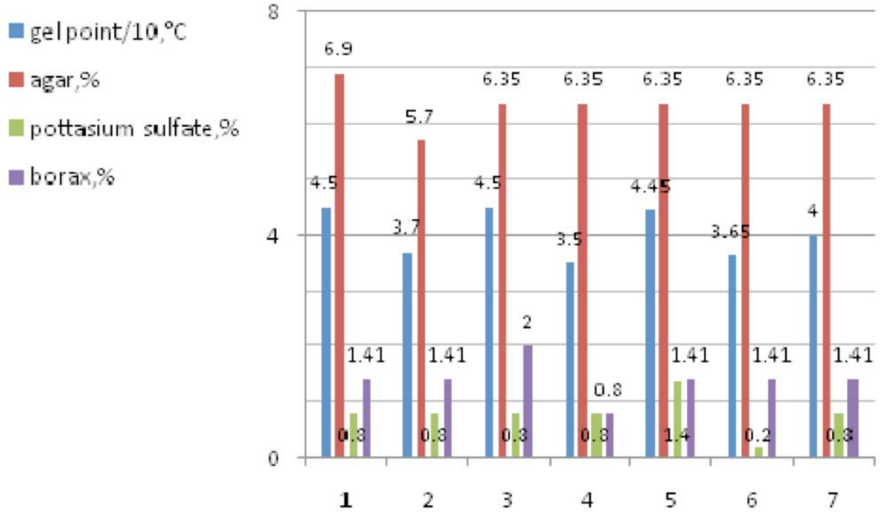

Figure 1: Effect of composition on Gel point of agar hydrocolloid impression.

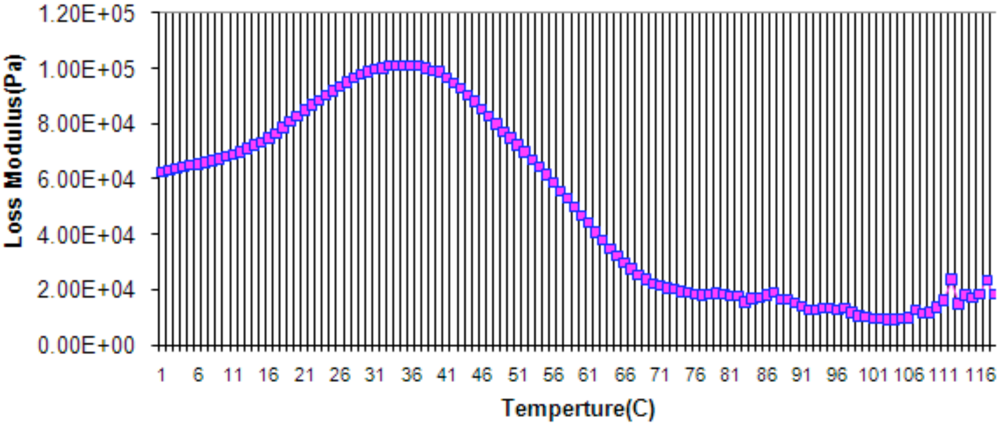

Figure 2: Modulus versus temperature for sample D.

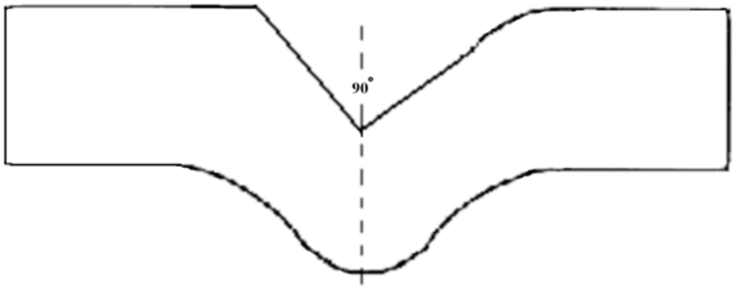

Figure 3: Dimensions of specimen for tear strength test.

Figure **3** shows test specimen used in ISO1564 (ANSI/ADA NO.11).

According to ISO1564 (ANSI/ADA NO.11) [7-8], it should be more than 0.5 N/mm when used as impression material. The result of tear testing is reported in Table **3** and Figure **4**.

Recovery from Deformation

After cooling the molds in room temperature, recovery from deformation has been determined. A dial indicator was used to measure the recovery. The height of cylindrical specimen is measured (h). After positioning the assembly on the specimen [2-3] the height of specimen is measured (h_1) after the material is compressed 20% for 1 second [7-8], the height of specimen was measured again (h_2). Five tests were carried out for each specimen.

Recovery form deformation is calculated by using Formula 2:

$$R = 100 \times \left(1 - \frac{h_1 - h_2}{h}\right) \tag{2}$$

According to ISO1564 (ANSI/ADA NO.11), it should be more than 96.5%. Test results are listed in Table **3**.

Compressive Strength

After cooling of cylindrical specimen in room temperature, compressive test was carried out. The Universal machine (Testometric, M350-5KN) was equipped with two flat plate. The compressive rate 254 mm/min [9] has been applied. Cylindrical specimen was placed between two plates and the maximum stress in longitudinal to crush the specimen is recorded. Another compression rate (49mm/min) [10] was used. The test was repeated for sample D at specified deformation rate (after testing, material was heated and poured in the molds again). According to standard requirement [2] compressive strength should be equal to 0.8 MPa.

Test results are listed in Table **4**. Proper deformation rate to evaluate compressive strength has been chosen based on other investigations. Some [10] researchers recorded that the best deformation rate is 0.8mm/s or 48 mm/min. According to others [9], deformation rate nearly 254 mm/min is suitable. The deformation rate of 48 mm/min is accepted because of

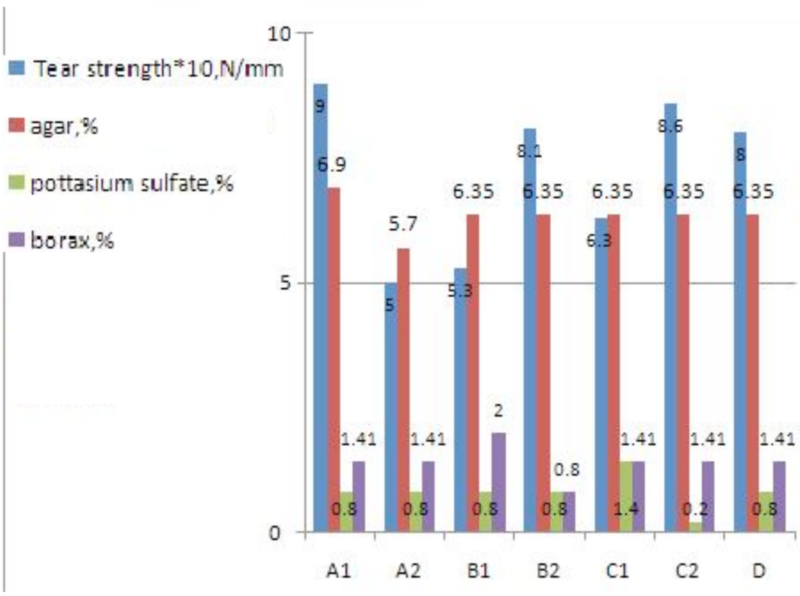

Figure 4: Effect of compositions of gels on tear strength.

maximum value obtained for compressive strength. Furthermore, removal rate of impression in dental laboratories is faster than 48 mm/min.

RESULTS AND DISCUSSION

Gel Temperature

According to ISO1564 (ANSI/ADA NO.11), the gelation temperature for different concentration of the agar has been compared. Sol-gel transition of reversible hydrocolloids depends on concentration of the agar as shown in Table **3** and Figure **1**. In other works [12], sol-gel transition temperature of agar has been compared by using three methods. Their used methods showed that gelation temperature increases with increasing of agar concentration [2].

In this work, gelation temperature changes *via* increasing of agar concentration from 35°C to 45°C which is confidence with standard value, between 37°C to 45°C [7].

Also gelation temperature is evaluated by rheological method. According to rheological method that represented by researchers, the onset of gelation is described by a very steep rise in the temperature dependence of the dynamic modulus [9]. In this study, the rheological method shows onset and maximum temperature at 37°C and 43°C respectively for sample D1. Figure **3** shows the rheological curve and test characteristic of sample D1. Comparing gelation temperature of samples D and D1 shows that nanosilver doesn't affect on the temperature obviously.

Tear Strength

According to ISO 1564(ANSI/ADA NO. 11), tear strength is sensitive to differences in concentration of components in formulation.

Comparing of A1, A2, D and D1 shows that concentration of the agar, as the base of gel, is enough to obtain optimum tear strength. Comparing of B1, B2, D and D1, shows that the concentration of borax is

Table 4: Compressive Strength in Different Deformation Rate (mm/min)

350mm/min	254mm/min	25.4mm/min	48mm/min	10mm/min	Deformation rate	
0.90	0.69	0.59	0.80	0.46	Compressive strength (Ave), (MPa)	18×20 (h ×d)mm
0.8-0.97	0.66-0.73	0.56-0.63	0.66-1.10	0.43-0.51	Min to Max (MPa)	
0.80	0.70	0.60	0.80	0.47	Compressive strength (Ave) (MPa)	16×20 (h ×d)mm
0.68-0.99	0.67-0.85	0.57-0.75	0.51-0.92	0.45-0.54	Min to Max (MPa)	

critical and it should be moderate. Addition of borax caused to increase strain, recovery from deformation and decrease tear strength in B1.

According to reports [13], gelation of agarose solutions has been thought to be due to coil–helix transition to form network cross-links according to Figures **5**, **6** [12, 14]. Therefore by increasing of the agar concentration, polymer chains per unit volume increase; increasing of entanglement between chains caused to increase strength of the gel.

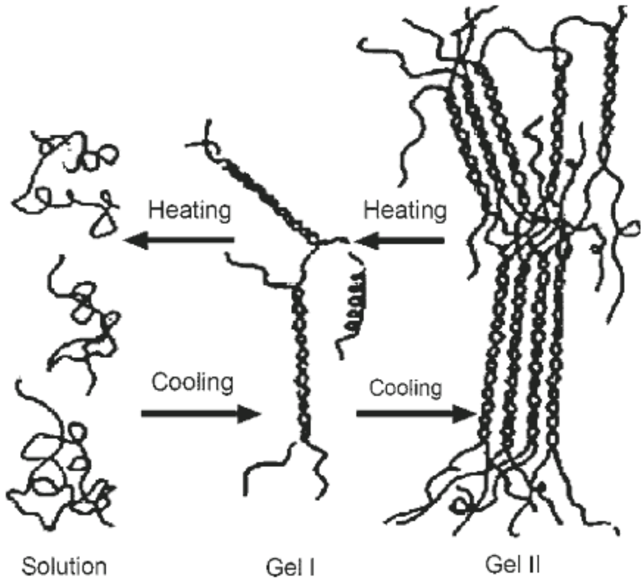

Figure 5: Mechanism of gel formation in agars due to hydrogen bonding, which forces double helix formation.

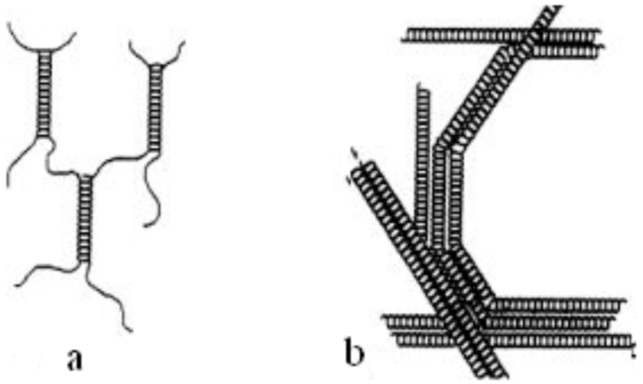

Figure 6: schematic drawing of possible mechanism of network formation in helical gels: gelation on the helical (**a**) and superhelical (**b**) levels.

Borax hydrolyzes in water to produce boric acid and the OH$^-$ ions can assist to hydrogen binding according to following steps [15]:

The boric acid further reacts with water to form a borate anion as follows:

$$B_4O_7^{-2}(aq) + 7H_2O \rightarrow 4H_3BO_3(aq) + 2OH^-(aq)$$

$$H_3BO_3(aq) + 2H_2O \rightarrow B(OH)_4^-(aq) + H_3O^+(aq)$$

The tetrahedral borate ion brings about hydrogen bonding between agar fibers to make fibril structure and then crosslink (Figure **5**).

In low amount of borax this phenomenon caused to strength of gel but in higher amounts, it may cause to decrease strength of gel because of limited possibility of hydrogen bonding and acts as spacer between fibers and finally decreases tear strength. Comparing of samples D, D1 shows that addition of nanosilver doesn't affect on tear strength, even maybe strengthen the gel.

Recovery from Deformation

Comparing of samples B1, B2, D and D1 shows that fluctuation of borax concentration hasn’t significant effect on recovery in equal proportions of the agar and potassium.

Comparing of samples D, D1 shows that nanosilver doesn't affect on recovery significantly. Comparing of samples A1, A2 with ISO1564 (ANSI/ADA NO.11), shows that concentration of agar has the most effective parameter on recovery from deformation of gels.

In samples A1, A2, by increasing agar in equal proportions of borax, recovery from deformation is increased but in samples A2 and B2, increasing in agar doesn't increase recovery from deformation and recovery from deformation of sample B2 is higher than sample A2, because of increasing borax in sample B2. The reason of this behavior discussed in previous section (Tear strength).

Compressive Strength

Although compressive strength test isn't characteristic property in ISO 1564(ANSI/ADA NO 11) standard, but according to dental material references it is important. According to Table **3**, the concentration of agar affects on compression strength. Comparison of samples A1, A2 shows that by adding of agar concentration, compressive strength is being increased [9] but comparison of sample A1, A2 and D, there is a significant difference between them. The concentration of borax can affect on compressive strength.

Comparison of samples C1, C2 shows that compressive strength increases in constant level of agar and borax by increasing of potassium sulfate. But comparison of sample C1, C2 and D, in constant level of agar and borax, the concentration of potassium sulfate can affect on compressive strength.

Comparison of samples B1, B2 shows that compressive strength increases in constant level of agar and potassium sulfate by increasing of borax. But comparison of sample B1, B2 and D, in constant level of agar and potassium sulfate, the concentration of borax can affect on compressive strength. Another investigation on compressive strength is study on change in deformation rate. In Table **4**, change in deformation rate caused to change in compressive strength. Table **4**, emphasize the importance of removing the impressions rapidly [2] or snap removal of agar hydrocolloid impression is recommended. Comparison of D, D1 shows that there isn't any significance difference between them. Concentration of material in agar impression composition is very important and it should consider all of requirements. For example, sample A1 is acceptable but sol-gel temperature transition is high. Sample C2 shows excellent tear strength but it is weak in recovery from deformation and other properties. Therefore optimum formulation is sample D1 same as D.

CONCLUSION

Sol-gel transition temperature of hydrocolloid depends on agar concentration. However higher concentration of potassium sulfate and borax caused to higher gel temperature. Increasing of concentration of potassium sulfate and borax caused to form gel in higher temperature and at higher concentration, they act as inhibitor for chain involvement as a spacer. Obtained gelation temperature by ISO test method and rheological method are comparable. To use of 500 ppm silver nanoparticles in agar impression formulation, mechanical properties don't change considerably and all of requirement is available and standard specification is passed. By using nanosilver in formulation disinfection process which caused to reduce inaccuracy of agar hydrocolloid impression, can be eliminated.

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