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ABSTRACT

The aim of this study was to produce composite blocks (CB) for CAD/CAM applications by high-temperature-pressure (HT/HP) polymerization of resin-infiltrated glass-ceramic networks. The effect of network sintering and the absence/presence of initiator was investigated. Mechanical properties were determined and compared with those of Paradigm MZ100 (3M ESPE) blocks and HT/HP polymerized experimental “classic” CB, in which the filler had been incorporated by conventional mixing. The networks were made from glass-ceramic powder (VITA Zahnfabrik) formed by slip casting and were either sintered or not. They were silanized, infiltrated by urethane dimethacrylate, with or without initiator, and polymerized under HT/HP (300 MPa, 180°C) to obtain resin-infiltrated glass-ceramic network (RIGCN) CB. HT/HP polymerized CB were also made from an experimental “classic” composite. Flexural strength (σ_f), fracture toughness (K_{IC}), and Vickers hardness were determined and analyzed by one- or two-way analysis of variance (ANOVA), Scheffé multiple-means comparisons ($\alpha = 0.05$), and Weibull statistics (for σ_f). Fractured surfaces were characterized with scanning electron microscopy. The mechanical properties of RIGCN CB were significantly higher. Sintering induced significant increases in σ_f and hardness, while the initiator significantly decreased hardness. The results suggested that RIGCN and HT/HP polymerization could be used to obtain CB with superior mechanical properties, suitable for CAD/CAM applications.

KEY WORDS: composite materials, CAD, glass-ceramics, polymers, materials science, prosthodontics.

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High-temperature-pressure Polymerized Resin-infiltrated Ceramic Networks

INTRODUCTION

Chairside computer-aided design/manufacturing (CAD/CAM) is currently gaining rapid popularity (Van Noort, 2012) because it allows the dentist to eliminate the need for a technician for certain laboratory steps, to obtain a constant quality of work, and to shorten the fabrication process. CAD/CAM also enables new materials to be used with better properties compared with those used in direct restorative procedures.

Thanks to their chemical stability, ceramics have good mechanical and optical properties and excellent biocompatibility. However, once they are in place, subsequent interventions may be problematic. Resin composites are easier to machine, but their wear, mechanical properties, and biocompatibility are inferior to those of ceramics. Consequently, it would be desirable to develop resin composite blocks (CB) with enhanced properties, suitable for CAD/CAM applications. For this, new industrial processes could be used. The use of high-temperature/high-pressure (HT/HP) polymerization (300 MPa, 180°C) (Sadoun, 2012; Nguyen *et al.*, 2012, 2013; Franco Steier *et al.*, 2013) and of interpenetrating network technology (Sadoun, 2011; He and Swain, 2011; Coldea *et al.*, 2013) significantly enhanced the mechanical properties of composites and could probably decrease the release of monomers. Moreover, the possibility of achieving a higher volume fraction filler (V_f) could also increase flexural strength (σ_f), modulus of elasticity, and hardness while decreasing polymerization shrinkage, thermal expansion coefficient, absorption coefficient, and water solubility (Bowen, 1963, 1964; Li *et al.*, 1985; Braem *et al.*, 1989; Shajii and Santerre, 1999; Chun *et al.*, 2009). However, increasing V_f makes mixing more difficult and promotes the formation of voids and poor filler distribution, with clusters, and consequently could lead to a decrease in σ_f (Atsuta and Turner, 1982; Ilie and Hickel, 2009). To solve this problem associated with the mixing of fillers into the monomers, another method of incorporating fillers should be considered.

During the 1980s, to enhance the mechanical properties of ceramics, investigators were successful in increasing the ratio of crystalline/glass phase to achieve full-ceramic crowns. The first interpenetrating networks developed for dental materials were the glass-infiltrated ceramics, called In-Ceram[®] (Vita Zahnfabrik, Bad Säckingen, Germany). The process led to ceramics with higher alumina content ($V_f = 75\%$) with enhanced mechanical properties compared with those of (40-50)% V_f leucite-containing ceramics. In dental composites, V_f could be increased beyond the current limits achievable by mixing by using the In-Ceram[®] process.

Sintered alumina networks vacuum-infiltrated with monomers and polymerized at 0.1 MPa have already been manufactured (Giordano, 1997, 1998; Chaiyabutr *et al.*, 2009). The 1.78 refractive index of alumina compared with the 1.48 to 1.53 refractive index of methacrylate monomers leads to an

Table 1. Details of Materials Used[#]

Composite and Manufacturer	Matrix ^{&}	Filler	V _f (%)	Configuration Filler	Initiator	Group	Polymerization Parameters
PARADIGM MZ100 (3M ESPE)	Bis GMA TEGDMA	Zirconia Silica 0.6 µm	64.2	Mixing with matrix		P	As-received CAD/CAM blocks
Experimental	100% UDMA	VITA Mark II	73.8	Sintered block, 800°C	Di-tert-amyl peroxide	ESI	HT/HP
Experimental	100% UDMA	VITA Mark II	73.8	Sintered block, 800°C	None	ES	HT/HP
Experimental	100% UDMA	VITA Mark II	72.6	Non-sintered block	Di-tert-amyl peroxide	EI	HT/HP
Experimental	100% UDMA	VITA Mark II	72.6	Non-sintered block	None	E	HT/HP
Experimental	100% UDMA	VITA Mark II	65.0	Mixing with matrix	Di-tert-amyl peroxide	EM	HT/HP

[#]The composition of the organic matrix and filler content of Paradigm MZ100 was obtained from manufacturer's data.

[&]BisGMA = bisphenol A glycol-dimethacrylate; TEGDMA = triethyleneglycol dimethacrylate; UDMA = urethane dimethacrylate (CAS: 72869-86-4); VITA Mark II = feldspathic ceramic reinforced with albite nepheline.

opaque microstructure; using a glass-ceramic would be a better compromise between mechanical and optical properties. Enamic blocks (VITA Zahnfabrik), based on this principle, became commercially available in early 2013. Furthermore, monomer polymerization leads to volume shrinkage and internal stress (Ferracane, 2005; Park and Ferracane, 2006) and, in the case of a rigid sintered network, could lead to interfacial debonding between polymer and ceramic network. Polymerization under HP could limit shrinkage by reducing free volume (Brosh *et al.*, 2002; Kaminski *et al.*, 2008; Kwiatkowski *et al.*, 2008; Schettino *et al.*, 2008) and could also limit the development of internal stress (Sadoun, 2011).

The aim of this study was to produce and characterize experimental CB, with V_f higher than 70%, based on resin-infiltrated glass-ceramic networks (RIGCN), polymerized under HT/HP, suitable for CAD/CAM applications. Their mechanical properties were compared with those of a commercially available dental CAD/CAM CB and with those of HT/HP polymerized experimental "classic" CB, in which the filler had been incorporated by conventional mixing. The influence of the status of the network (sintered or not) and the absence or presence of initiator was also investigated.

The null hypotheses tested were: (1) There are no differences in the mechanical properties among HT/HP polymerized RIGCN CB, a commercially available dental CAD/CAM CB, and an experimental "classic" CB; (2) sintering does not affect the mechanical properties of RIGCN CB; and (3) the initiator does not affect the mechanical properties of RIGCN CB.

MATERIALS & METHODS

Four RIGCN CB were manufactured under HP/HT conditions. As controls, we used experimental "classic" CB, in which the filler had been incorporated by conventional mixing, polymerized under the same HT/HP conditions, and a commercially available CAD/CAM CB, Paradigm MZ100 (3M ESPE, St. Paul, MN, USA). Table 1 summarizes the details regarding the materials used and the group codes; sample preparation and characterization procedures are described below.

Processing of the Glass-Ceramic Network

The inorganic network was processed by slip casting with glass-ceramic powder VITA Mark II (VITA Zahnfabrik) with 5.13 µm D50 grain size distribution. We produced the slip by mixing 56% volume ratio glass-ceramic powder with water in a planetary mixer (Thinky ARE-250, Thinky Corporation, Tokyo, Japan). The slip was cast in a plaster mold to achieve the grain agglomeration and then was dried overnight at room temperature. The non-sintered blocks were dehydrated in a furnace at 160°C for 2 hrs. We added a sintering step at 800°C for 2 hrs to obtain partially sintered blocks with open porosities (0.25 µm). The sizes and volumes of porosities were measured with a mercury porosimeter (Autopore, Micromeritics, Norcross, GA, USA).

Monomer Infiltration of Open-porosity Networks

Sintered and non-sintered porous blocks were silanated with pre-hydrolyzed 3-(trimethoxysilyl)propyl methacrylate (Sigma-Aldrich, St. Louis, MO, USA) and then heated at 140°C for 6 hrs. Complete vacuum infiltration was done with pure urethane dimethacrylate (UDMA) (Esstech, Essington, PA, USA) (no initiator) or with a mixture of 99% (wt) UDMA and 1% (wt) initiator (di-tert amyl peroxide (Sigma-Aldrich); after vacuum release, the infiltrated blocks were maintained at 70°C for 8 hrs.

Preparation of Experimental "Classic" Composite

The same glass-ceramic powder, VITA Mark II, was used as filler in UDMA monomer, along with di-tert amyl peroxide as initiator. The incorporation of filler into the monomer was accomplished by the use of a planetary mixer to obtain the experimental "classic" composites with 65% V_f.

Thermo-polymerization under High Pressure

A differential scanning calorimeter (DSC823, Mettler-Toledo, Greifensee, Switzerland) was used to determine the thermo-polymerization temperature of UDMA without initiator at atmospheric pressure. Since the determined polymerization temperatures were in the 160 to 180°C range, it was decided to

conduct all polymerization reactions at 180°C. Thereafter, approximately 100 g of the experimental “classic” composite placed inside a flexible silicone tube (25-mm internal diameter) and the sintered/non-sintered infiltrated blocks were introduced into a custom-built autoclave, with pressure and temperature control (LabVIEW 8.6, National Instruments, Austin, TX, USA). A thermocouple was placed in the proximity of the sample to facilitate accurate monitoring and, *via* feed-back, control of the temperature. In the first step, the pressure within the autoclave was increased to 300 MPa at a rate of 1 MPa/sec. In the second step, the temperature was increased to 180°C at a rate of 2°C/min. The sample was maintained at 300 MPa and 180°C for 60 min before being cooled and the pressure released.

Flexural Strength

One part of each HT/HP polymerized CB was cut, with an Isomet saw (Buehler, Lake Bluff, IL, USA) under water irrigation, into 30 rectangular bars (4 x 2 x 20 mm). Each bar was wet-polished on a 4,000-grit silicon carbide (SiC) paper disc, and its dimensions were measured with a digital caliper (Mitutoyo Co., Kawasaki, Japan) before being tested. The bars were loaded in a three-point bending device (16-mm span) at a cross-head speed of 1 mm/min, in a computer-controlled (NexyGen®, Lloyd, UK) Lloyd LRX (Lloyd, UK) universal testing machine.

Flexural strength, σ_f , was calculated according to the formula:

$$\sigma_f = \frac{3FL}{2hc^2}$$

where F is the load at fracture, L the span, h the specimen width, and c the specimen height.

Fracture Toughness

One part of each HT/HP polymerized CB was cut, with an Isomet saw under water irrigation, into 8 rectangular bars (8 x 8 x 15 mm), which were then wet-ground on 800-grit SiC to obtain 6 x 6 x 6 x 12 mm equilateral triangular prisms. Fracture toughness (K_{IC}) was determined with the notchless triangular prism (NTP) specimen K_{IC} test (Ruse *et al.*, 1996). The prisms were secured into one half of the specimen holder, and a sharp scalpel was used to create a small (< 0.1-mm-deep) defect along the loading edge, before it was secured to the second half of the specimen holder. The specimens were loaded in tension, in a computer-controlled (Bluehill, Instron Canada Inc., Burlington, ON) universal testing machine (Instron model 4301), at a cross-head speed of 0.01 mm/min until crack arrest or fracture. The maximum load recorded (P_{max}) was used to calculate K_{IC} in MPa·m^{1/2} according to the following equation, proposed by Barker (1977) and adopted by ASTM standard E1304:

$$K_{IC} = \frac{Y_{min}^* P_{max}}{DW^{1/2}}$$

where Y_{min}^* is the minimum dimensionless stress intensity coefficient, estimated at 28 for NTP samples (Ruse *et al.*, 1996), D the specimen-holder diameter (12 mm), and W the specimen-holder length (10.4 mm).

Hardness

To improve reading, test samples of each material were surface-coated with a thin gold layer by means of a sputter-coater (SC500, Bio-Rad, Maxted Road, Hemel Hempstead, Herts, UK). Surface microhardness was measured by means of a Vickers indenter (MH3, Metkon, Bursa, Turkey), under a 10-N loading and 20-second dwell time. Thirty determinations on 5 samples were made for each material.

Fractured Surface Morphology

Representative fractured NTP specimens of each material were sputter-coated with gold. The surface morphology was then characterized under a scanning electron microscope (SEM) (JSM-6400, JEOL Ltd., Tokyo, Japan) at low and high magnification.

Statistical Analysis

The results were analyzed by one- or two-way analysis of variance (ANOVA) followed, if warranted, by Scheffé multiple-mean comparisons ($\alpha = 0.05$), using PASW Statistics 18 (SPSS, Chicago, IL, USA). Weibull statistics parameters were calculated for the σ_f data by the Weibull statistics option in Excel® (Microsoft, USA). The description of the Weibull distribution is given by

$$P_f = 1 - e^{-\left(\frac{\sigma}{\sigma_0}\right)^m}$$

where m is the shape parameter (Weibull modulus), σ_0 is the scale parameter or characteristic strength $\sigma_{63.21\%}$ (Weibull, 1951; Bona *et al.*, 2003). P_f , the fracture probability, is defined by the relation

$$P_f = \frac{\kappa}{N + 1}$$

where κ is the rank in strength from least to greatest, and N denotes the total number of specimens.

RESULTS

The results of the mechanical characterizations are summarized in Table 2, along with the results of the statistical analysis.

The results have shown that RIGCN CB obtained *via* HP/HT polymerization (groups ES, ESI, E, EI) have σ_f , $\sigma_{63.21\%}$, hardness, K_{IC} , and Weibull modulus superior to those of Paradigm MZ100 (group P) and to those of the experimental “classic” CB (group EM). The first null hypothesis was therefore rejected.

Two-way ANOVA tables for σ_f , hardness, and KIC are shown in Table 3 for sintered/non-sintered and with/without initiator RIGCN CB. The analysis showed that sintering resulted in significantly increased σ_f and hardness ($p < .05$). The Weibull modulus, however, was higher for the non-sintered networks. Significant differences ($p < .05$) were noted in the effect of initiator on hardness, which decreased with the use of initiator. Significant differences ($p < .05$) were also noted in the sintering and initiator combined effect for σ_f and hardness. Highest σ_f was obtained with sintered and initiator and highest hardness with sintered with no initiator.

In view of the above, the second and third null hypotheses were rejected.

Table 2. Results (mean $\pm$ SD) of Mechanical Characterization and Statistical Analysis[#]

Group ^{\$}						
Property	ESI	ES	EI	E	EM	P
σ_f (in MPa)	305.2 $\pm$ 53.7 ^a	288.3 $\pm$ 39.6 ^{ab}	256.1 $\pm$ 40.3 ^c	270.9 $\pm$ 26.1 ^{bc}	121.8 $\pm$ 23.17 ^d	138.2 $\pm$ 24.3 ^d
Hardness (in HVN)	137.8 $\pm$ 5.1 ^b	144.6 $\pm$ 3.2 ^a	122.7 $\pm$ 7.6 ^c	136.5 $\pm$ 5.5 ^b	101.9 $\pm$ 3.5 ^e	114.8 $\pm$ 4.3 ^d
K_{IC} (in MPa·m ^{1/2})	2.7 $\pm$ 0.5 ^a	2.8 $\pm$ 0.7 ^a	3.1 $\pm$ 1 ^a	2.9 $\pm$ 1 ^a	1.8 $\pm$ 0.6 ^b	0.8 $\pm$ 0.2 ^c
Weibull modulus	6.4	8.11	11.7	11.5	5.8	5.3
* $\sigma_{63.21\%}$ (in MPa)	327.2	305.6	273	282.7	131.2	149.3

[#]Superscript letters indicate statistically homogeneous subgroups within a material category (Scheffé test, $\alpha = 0.05$) The same superscript letters demonstrate no significant differences in each line.

^{\$}ESI = experimental composite, sintered, with initiator, polymerized under HT/HP; ES = experimental composite, sintered, without initiator, polymerized under HT/HP; EI = experimental composite, not sintered, with initiator, polymerized under HT/HP; E = experimental composite, not sintered, without initiator, polymerized under HT/HP; EM = control experimental classic composite (filler incorporated into the matrix by mixing), polymerized under HT/HP; P = paradigm MZ100.

*Weibull characteristic strength.

Table 3. Two-way Analysis of Variance (ANOVA) for Mechanical Properties

Flexural Strength (in MPa)							
Factor		Mean $\pm$ SD	df	Type III Sum of Squares	Mean Square	F	Significance
S: Sintering	sintered	296.6 $\pm$ 47.5	1	37,484.921	37,484.921	21.992	*
	non-sintered	263.6 $\pm$ 34.4					
I: Initiator	initiator	281.7 $\pm$ 53.4	1	37.493	37.493	0.022	–
	no initiator	280 $\pm$ 34.7					
S $\times$ I	sintered initiator	305.2 $\pm$ 53.7	1	8,433.128	8,433.128	4.948	*
	non-sintered initiator	288.3 $\pm$ 39.6					
	non-sintered no initiator	256.1 $\pm$ 40.3					
	no initiator	270.9 $\pm$ 26.1					
Hardness (in HVN)							
Factor		Mean $\pm$ SD	df	Type III Sum of Squares	Mean Square	F	Significance
S: Sintering	sintered	141.2 $\pm$ 5.4	1	4,124.753	4,124.753	131.379	*
	non-sintered	129.4 $\pm$ 9.6					
I: Initiator	initiator	130.1 $\pm$ 9.9	1	3,271.967	3,271.967	104.217	*
	no initiator	140.6 $\pm$ 6					
S $\times$ I	sintered initiator	137.8 $\pm$ 5.1	1	372.347	372.347	11.86	*
	non-sintered initiator	144.6 $\pm$ 3.2					
	non-sintered no initiator	122.7 $\pm$ 7.6					
	no initiator	136.4 $\pm$ 5.5					
Fracture toughness (in MPa·m ^{1/2})							
Factor		Mean $\pm$ SD	df	Type III Sum of Squares	Mean Square	F	Significance
S: Sintering	sintered	2.8 $\pm$ 0.6	1	0.061	0.061	0.1	–
	non-sintered	3 $\pm$ 1					
I: Initiator	initiator	2.9 $\pm$ 0.7	1	0.648	0.648	1.056	–
	no initiator	2.8 $\pm$ 0.8					
S $\times$ I	sintered initiator	2.7 $\pm$ 0.5	1	0.116	0.116	0.189	–
	non-sintered initiator	2.8 $\pm$ 0.7					
	non-sintered no initiator	3.1 $\pm$ 1					
	no initiator	2.9 $\pm$ 1					

*Significant difference was detected ($p < .05$).

Figure. SEM micrographs of the fracture surfaces: (a) experimental composite, sintered, with initiator, polymerized under HT/HP (ESI); (b) experimental composite, sintered, without initiator, polymerized under HT/HP (ES); (c) experimental composite, not sintered, with initiator, polymerized under HT/HP (EI); (d) experimental composite, not sintered, without initiator, polymerized under HT/HP (E); (e) control experimental classic composite (filler incorporated into the matrix by mixing), polymerized under HT/HP (EM); (f, g) paradigm MZ100 (P).

SEM characterizations of fractured K_{IC} specimens from all the materials showed the presence of fewer and smaller voids and rougher surfaces in HT/HP RIGCN CB.

DISCUSSION

Compared with EM and P, the higher V_f (8% to 9% more) of RIGCN CB could explain the significant increase in σ_p , $\sigma_{63.21\%}$, K_{IC} ,

and hardness, regardless of the status of the network (sintered or not-sintered) or whether or not initiator was used.

The small increase in V_p from 72.6% to 73.8%, noted in RIGCN CB with sintered networks, due to the shrinkage caused by the sintering process, along with the presence of sintering necks, could explain the observed higher σ_f and hardness in comparison with those of non-sintered networks. This concurs with a previous study that showed increased elastic modulus and hardness of CB with increasing degree of sintering (Coldea *et al.*, 2013).

Even higher V_f could be reached (in the 80% range) by optimizing the particle size distribution and increasing the temperature and sintering time. However, the necessity to maintain an adequate open porosity for monomer infiltration limits V_f . Moreover, the more sintered the networks are, the more sintering necks exist, rendering the network harder and less machinable.

It is thus possible to modulate the properties of the network by changing the grain size distribution, the green density, and the sintering parameters (time, temperature). If one desires, a more rigid and denser network with higher flexural modulus could be obtained by increasing sintering time and temperature.

Infiltrating monomers into a porous glass-ceramic network allowed us to overcome the limit of incorporating filler by mixing. Indeed, mixing at 65% V_f has already resulted in a viscous and heterogeneous paste, and even with high-pressure polymerization, the presence of many defects was reported to cause a decrease of σ_f (Atsuta and Turner, 1982; Nguyen *et al.*, 2013).

There was no significant difference in K_{IC} between RIGCN CB with a sintered and non-sintered network. The lack of a rigid network may have increased the energy-absorbing capability in the vicinity of the crack tip, thus explaining the good K_{IC} of RIGCN CB with non-sintered networks.

Weibull moduli of RIGCN CB were higher than those of P and EM, and, in association with the results of SEM characterizations, it may be concluded that the fabrication process results in fewer and smaller flaws. Higher Weibull moduli of RIGCN CB with a non-sintered network could be explained by the absence of the sintering necks, which would permit the movement of particles before polymerization under pressure to reduce the flaw size distribution.

The presence of an initiator did not significantly enhance σ_f or K_{IC} , while hardness of RIGCN CB was decreased significantly. Therefore, the use of an initiator does not seem necessary under HT/HP polymerization conditions, especially since its absence could also improve the biocompatibility.

SEM micrographs of fractured surfaces (Fig.) showed interfacial cracks and flaws for Paradigm. Cracks were noted in the matrix for RIGCN CB obtained *via* HT/HP, and there were fewer and smaller voids. High pressure is an important parameter for the manufacture of RIGCN CB, since it facilitates compensation for the polymerization shrinkage of the infiltrated resin and control of the interfacial stress, and improves bonds between filler and polymers.

Furthermore, fractured surfaces of RIGCN CB obtained *via* HT/HP are rougher than those of EM and P, which indicated higher fracture energy for RIGCN CB and therefore could explain the better K_{IC} .

The results of this study showed that the mechanical properties of RIGCN CB obtained *via* HT/HP polymerization were significantly superior to those of a commercial CB and those of an experimental “classic” CB obtained *via* HT/HP and that higher V_f and the presence of a sintered network significantly increased σ_f and hardness.

The results suggest that resin CB with superior mechanical properties could be obtained *via* HT/HP polymerization of resin-infiltrated glass-ceramic networks, and that they could be suitable for CAD/CAM applications. Further studies focusing on wear, monomer release, biocompatibility, etc., are necessary before clinical trials can be proposed.

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- The presence of an initiator does not significantly enhance σ_f or K_{IC} , while hardness of RIGCN CB was decreased significantly. Therefore, the use of an initiator does not seem necessary under HT/HP polymerization conditions, especially since its absence could also improve the biocompatibility.
- SEM micrographs of fractured surfaces (Fig.) showed interfacial cracks and flaws for Paradigm. Cracks were noted in the matrix for RIGCN CB obtained via HT/HP, and there were fewer and smaller voids. High pressure is an important parameter for the manufacture of RIGCN CB, since it facilitates compensation for the polymerization shrinkage of the infiltrated resin and control of the interfacial stress, and improves bonds between filler and polymers.
- Furthermore, fractured surfaces of RIGCN CB obtained via HT/HP are rougher than those of EM and P, which indicated higher fracture energy for RIGCN CB and therefore could explain the better K_{IC} .
- The results of this study showed that the mechanical properties of RIGCN CB obtained via HT/HP polymerization were significantly superior to those of a commercial CB and those of an experimental “classic” CB obtained via HT/HP and that higher V_f and the presence of a sintered network significantly increased σ_f and hardness.
- The results suggest that resin CB with superior mechanical properties could be obtained via HT/HP polymerization of resin-infiltrated glass-ceramic networks, and that they could be suitable for CAD/CAM applications. Further studies focusing on wear, monomer release, biocompatibility, etc., are necessary before clinical trials can be proposed.
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