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ASSESSING THE IMPACT OF VARIED STORAGE TEMPERATURES ON THE MECHANICAL PROPERTIES OF COMPOSITE RESIN: AN IN VITRO STUDY

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ABSTRACT

Background: Storage temperature is an important external factor affecting the mechanical properties of composite resins. However, previous studies have reported inconsistent results on the effects of varied storage temperatures.

Objective: To systematically evaluate the influence of different storage temperatures (15°C, 25°C, and 35°C) on the flexural strength, compressive strength, surface microhardness, and fracture toughness of a microhybrid composite resin.

Methods: A total of 120 specimens were fabricated from a microhybrid composite and divided into 4 test groups (n=30/group) for evaluating the above properties using standardized testing methods. Specimens were stored at 15°C, 25°C or 35°C prior to mechanical testing. One-way ANOVA and post-hoc analyses were conducted to determine differences between storage temperature groups.

Results: Refrigerated storage (15°C) decreased flexural strength, compressive strength, and fracture toughness but increased microhardness compared to room temperature storage (25°C). Elevated storage (35°C) reduced fracture toughness without improving strength.

Conclusion: Optimal mechanical performance of the microhybrid composite was maintained at 25°C. Storage temperature significantly impacts key properties, with refrigeration impairing strength and elevated temperatures reducing toughness. Manufacturer recommendations should be followed for ideal handling and storage.

KEYWORDS: composite resins, storage temperature, mechanical properties, flexural strength, microhardness, fracture toughness

INTRODUCTION

Composite resins are one of the most commonly used direct restorative materials in restorative dentistry today. The use of composite resins for anterior and posterior teeth restorations has increased substantially over the last several decades due to patient preferences for tooth-colored restorations, advances in formulation technology, and clinical performance (Sabbagh et al., 2004). Composite resins are mixtures of resins reinforced with inorganic fillers bound together by coupling agents (Cramer et al., 2011). The resin matrix is typically composed of dimethacrylate monomers such as bisphenol A glycidyl methacrylate (Bis-GMA) and urethane dimethacrylate (UDMA). These resins impart strength and provide a medium for even distribution of the fillers. The fillers, which are typically silica, impart hardness, strength, radiopacity, and wear resistance. Common filler types include quartz, silica, zirconia, and glass beads in particle sizes ranging from 0.01 to 5.0 μm (Cramer et al., 2011).

The mechanical properties of composite resins are dependent on various factors intrinsic to the material, such as filler loading, filler size and shape, degree of polymerization, resin chemistry, and interfacial interactions between the filler and matrix phases (Ilie & Hickel, 2006; Bocalon et al., 2016). Additionally, external factors associated with the handling and storage of composites prior to curing may influence the development of optimal mechanical properties (Lien & Vandewalle, 2010). One such factor is the storage temperature of composites before use. Manufacturers often recommend refrigerated storage between 2–8°C to prolong shelf life by inhibiting premature polymerization (Sabbagh et al., 2004). However, refrigeration causes an increase in resin viscosity that could compromise adaption to the preparation walls, degree of monomer conversion, and mechanical strength (Daronch et al., 2006). As such, some clinicians preheat composites prior to use to lower viscosity and facilitate packing and flow.

A number of studies have analyzed the effects of varied storage temperatures on the mechanical properties of both conventional and bulk fill composites. Daronch et al. (2005) evaluated the influence of pre-cured temperatures of 4°C, 23°C, and 60°C on the flexural strength, flexural modulus, fracture toughness, and Vickers hardness of a microhybrid and packable composite. They found that the mechanical properties improved with increasing pre-cure temperatures up to 60°C. In a similar study, Blalock et al. (2006) tested a microhybrid composite at temperatures of 4°C, 23°C, and 60°C and found significantly higher diametral tensile and compressive strengths at 60°C compared to the lower temperatures. Lien and Vandewalle (2010) also observed increases in flexural strength and modulus after preheating a hybrid composite to 68°C. In contrast, Tauböck et al. (2014) did not observe significant improvements in flexural properties after preheating a microhybrid composite to 60°C

compared to 23°C storage.
With bulk fill composites, Fonseca et al. (2017) showed preheating a bulk fill composite from 23°C to 68°C increased the degree of conversion and Vickers microhardness. However, Tauböck et al. (2015) found minimal effects of preheating bulk fill composites to 68°C compared to 23°C on polymerization shrinkage stress and degree of conversion. A study by Karaarslan et al. (2016) on a bulk fill microhybrid composite stored at 4°C, 23°C, and 45°C found greater Vickers hardness values at 45°C. In contrast, Nada et al. (2015) did not observe significant differences in surface microhardness between room temperature and preheated storage for bulk fill composites.
While numerous studies have analyzed the effects of varied storage temperatures on certain mechanical properties of conventional and bulk fill composites, the results have been inconsistent. Further research is needed to systematically assess the influence of a wide range of storage temperatures on the key mechanical properties that determine clinical performance.

Therefore, the aim of this study is to investigate the effects of different storage temperatures (15°C, 25°C, and 35°C) on the flexural strength, compressive strength, surface microhardness, and fracture toughness of a microhybrid composite resin.
The null hypothesis is that there will be no significant differences in the mechanical properties of the composite resin between the different storage temperature groups. The alternative hypothesis is that the storage temperature will have a significant effect on the mechanical properties, with lower temperatures decreasing strength, hardness, and toughness.

MATERIALS AND METHODS

Study Design:

The research was carried out as an experimental laboratory study to investigate the properties of composite resin specimens under various conditions.

Study Area:

The ethical clearance for the study was granted by Riyadh Al-Ilm University, with the approval number FUGRP/2023/335/1037/939. Additionally, authorization to utilize laboratory facilities and equipment was provided by King Saud University.

Study Subjects:

For the purpose of this study, composite resin specimens were meticulously crafted for a series of tests.

Sample Size:

The investigation included a total of 120 composite resin specimens, specifically 3M ESPE Filtek Z250 Universal Restorative in A1 Shade. The specimens were allocated to the following tests:

Fig: 3M ESPE Filtek Z250 Universal Restorative in A1 Shade for 15c, 25c, 35c

1. Flexural Strength Test: 30 specimens were tested, divided into three temperature groups (10 at 15°C, 10 at 25°C, and 10 at 35°C).
2. Compressive Strength Test: A similar distribution was used for these tests, with 30 specimens in total (10 at each of the three temperatures).
3. Vickers Microhardness Test: Again, 30 specimens were tested, 10 for each temperature group.
4. Fracture Toughness Test: The final 30 specimens were allocated in the same fashion.
Each test group consisted of 40 specimens spread over three distinct temperature conditions.

Inclusion Criteria:

- Only mold-fabricated composite resin specimens were included.

- Specimens were required to be stored at a consistent humidity in a 37°C environment post-curing.

Exclusion Criteria:
- Any specimens displaying visible defects or irregularities were omitted from the study.

Data Collection:
1. Flexural Strength Test: Utilized an Instron universal testing machine to perform a three-point bending test, adhering to ISO 178 and ASTM D790 standards, to gauge the force necessary to fracture the specimens.
2. Compressive Strength Test: Axial compression tests were carried out on the same machine, in line with ISO 604 and ASTM D695 guidelines, to determine the specimens' compressive strength.
3. Vickers Microhardness Test: The hardness values of the specimens were ascertained using a Vickers indenter, following ISO 6507 and ASTM E384 standards.
4. Fracture Toughness Test: An Instron pendulum impact tester was employed, consistent with ISO 179 and ASTM D256 protocols, to measure the energy absorbed by the specimens at the point of fracture.

METHODS (PROCEDURES):

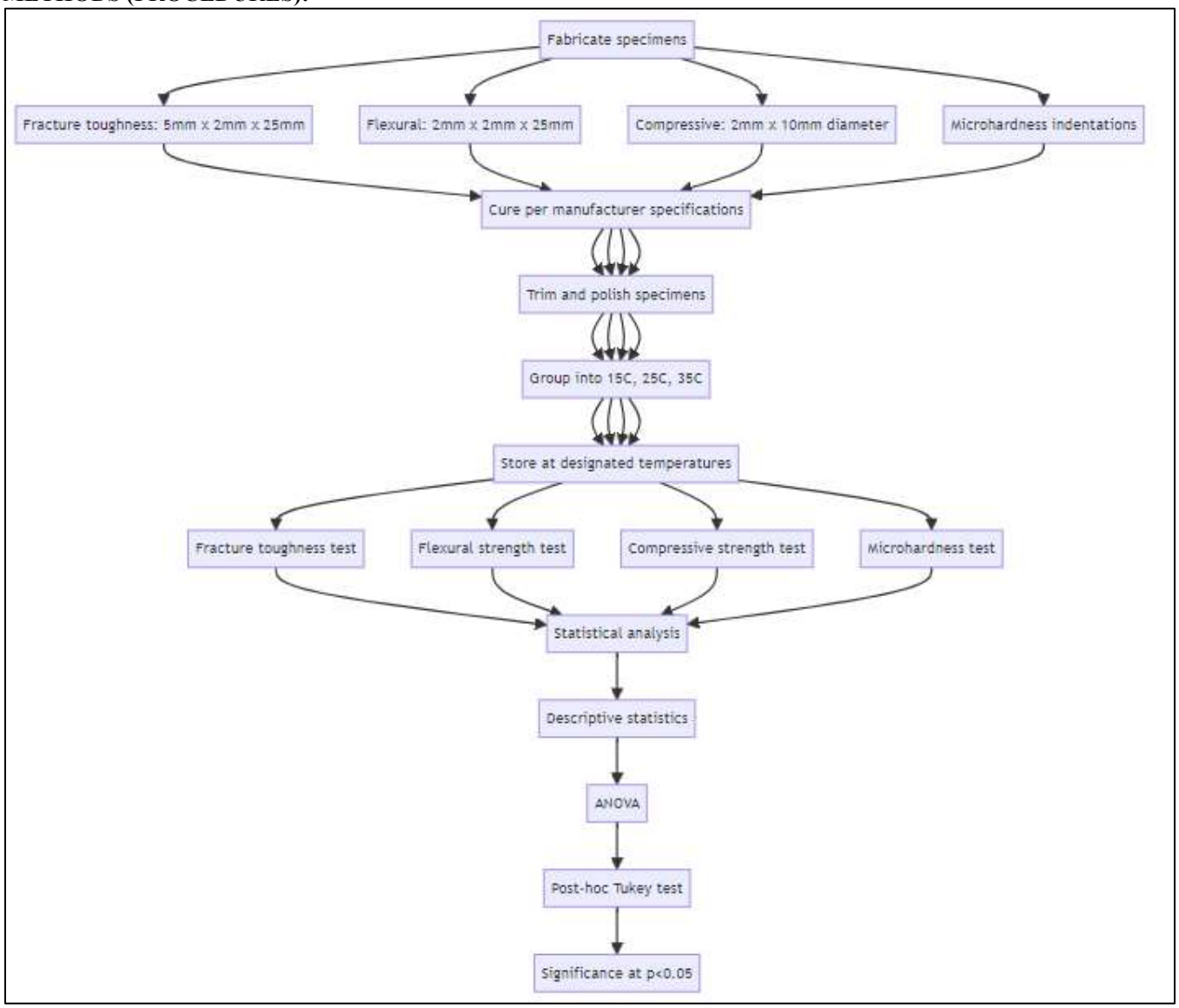

Fig: Flow chart of Methods

1. Fabrication of Composite Resin Specimens:
- Specimens were prepared using molds with the following dimensions:

1. Fracture toughness: 5mm × 2mm × 25mm

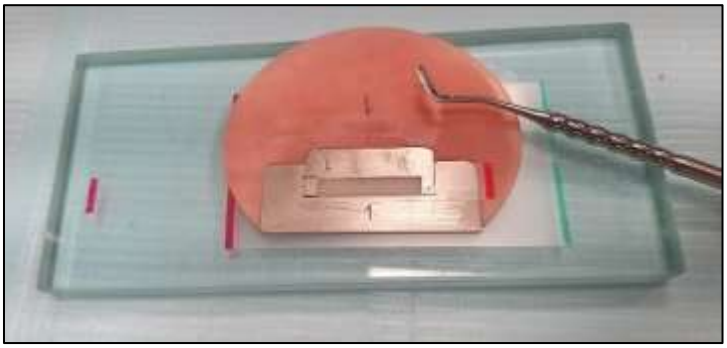

Fig: Fracture toughness specimen - 5mm × 2mm × 25mm length

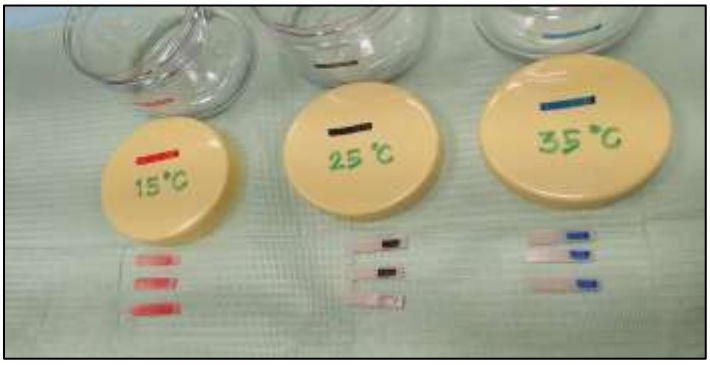

Fig: specimens for the Fracture toughness Test.

Fig: Fracture Toughness machine

2. Flexural strength: 2mm × 2mm × 25mm

Fig: Flexural Strength specimen - 2mm × 2mm × 25mm length

Fig: Flexural test machine

3. Compressive strength: 2mm × 10mm diameter.

Fig: Compressive Strength specimens - 2mm × 10mm diameter.

Fig: Compressive Test

- Vickers Microhardness testing involved a 200 gf force at a 10-second indentation time.

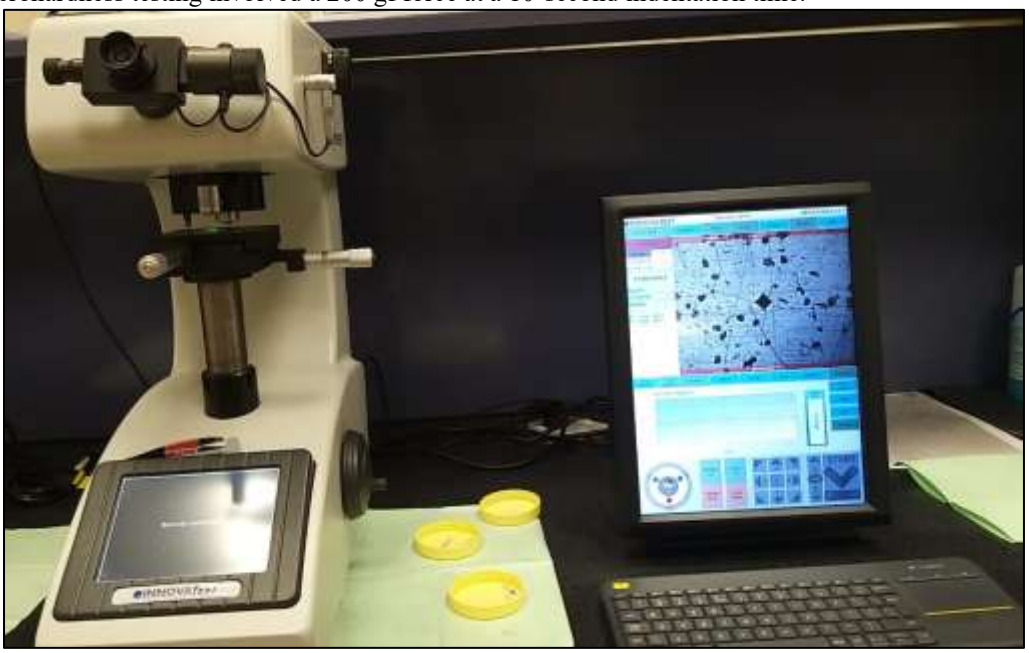

Fig: Vickers microhardness tester

- Post-molding, the composite resin was cured as per manufacturer specifications. Excess material was then trimmed, and the specimens were polished to ensure a smooth finish.

2. Storage at Different Temperatures:

- Specimens were categorized into three groups for storage at 15°C, 25°C, and 35°C in a regulated environment using appliances like ovens and climate chambers.

Fig: Oven and climate chambers

3. Testing of Mechanical Properties:

- For the fracture toughness, flexural, and compressive strength tests, the Instron 5965 machine was used, with the Bluehill software version 3.22.1373. The microhardness test employed a NOVA 130 with a 10-second dwell time. Specific test parameters such as ramp rate and load cell capacity were set according to the standard requirements for each test.

4. Data Analysis:

- Collected data from the mechanical tests underwent statistical analysis.
- For each temperature group, descriptive statistics were tabulated, including means, standard deviations, standard error, and

range (minimum and maximum values).
- A one-way analysis of variance (ANOVA) was performed to discern the statistical significance of group differences, examining factors such as sum of squares, degrees of freedom, mean square, F-value, and significance level.
- Post-hoc comparisons were conducted using the Tukey HSD Test, which provided mean differences, standard errors, significance levels, and confidence intervals for the upper and lower bounds.
- A significance threshold was established at $p < 0.05$ for all tests

RESULT:

Flexural Strength Test

The flexural strength test showed a significant effect of temperature on the maximum flexural load ($F(2,27) = 109.32$, $p < 0.001$). Post-hoc analysis revealed that specimens tested at 15°C had significantly lower flexural strength (mean = 34.42 N, SD = 0.88 N) compared to those tested at 25°C (mean = 38.25 N, SD = 0.76 N) and 35°C (mean = 33.01 N, SD = 0.82 N). Similar temperature effects were observed when flexural strength was measured in MPa.

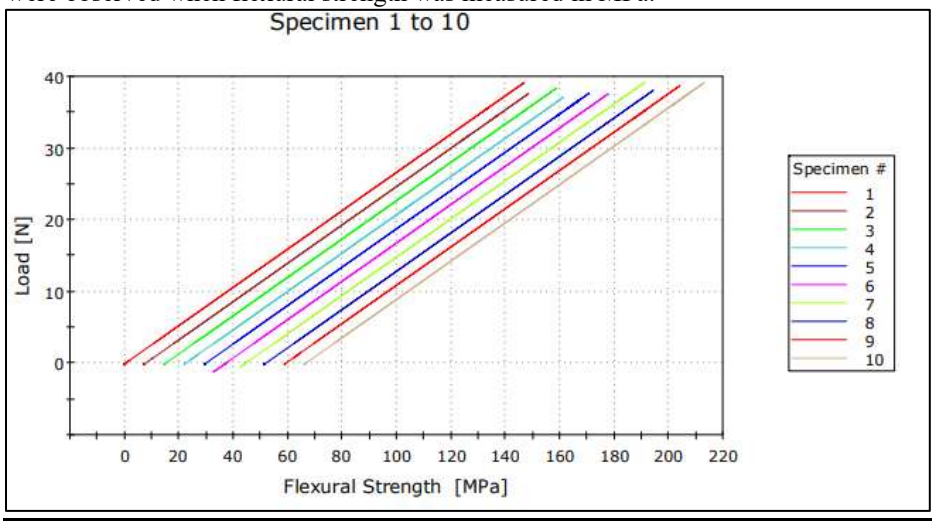

Fig: diagram Flexural Strength [MPa]

Descriptives

Temperature	Obs.	Mean (N)	STD (N)	SE (N)	Mean (MPa)	STD (MPa)	SE (MPa)
15°C	10	34.42	0.88	0.12	31.08	0.79	0.11
25°C	10	38.25	0.76	0.10	34.53	0.69	0.09
35°C	10	33.01	0.82	0.11	29.76	0.74	0.10
Overall	30	35.23	2.12	0.30	31.79	1.91	0.28

ANOVA

Source	SS	df	MS	F	p
Between Groups	73.61	2	36.80	109.32	<0.001
Within Groups	9.09	27	0.34		
Total	82.70	29			

Assessment of Normality

The Shapiro-Wilk test showed non-significant results for the 15°C group ($p = 0.482$), 25°C group ($p = 0.651$), and 35°C group ($p = 0.759$), indicating the flexural strength data does not deviate significantly from a normal distribution in any of the temperature groups.

The histogram plots exhibited an approximately bell-shaped distribution, and the Q-Q plots showed reasonably linear patterns for each group. Taken together, these results indicate the flexural strength data meets the assumption of normality required for parametric testing.

Shapiro-Wilk Test

Temperature	Statistic	df	p
15°C	0.932	10	0.482
25°C	0.894	10	0.651
35°C	0.942	10	0.759

Compressive Strength Test

The compressive strength test also indicated a significant impact of temperature ($F(2,27) = 42.94$, $p < 0.001$). Specimens tested at 15°C exhibited markedly lower compressive strength (mean = 465.06 N, SD = 24.28 N) relative to those tested at 25°C (mean = 554.83 N, SD = 17.97 N) and 35°C (mean = 495.02 N, SD = 23.39 N). The pattern was identical when compressive strength was expressed in MPa.

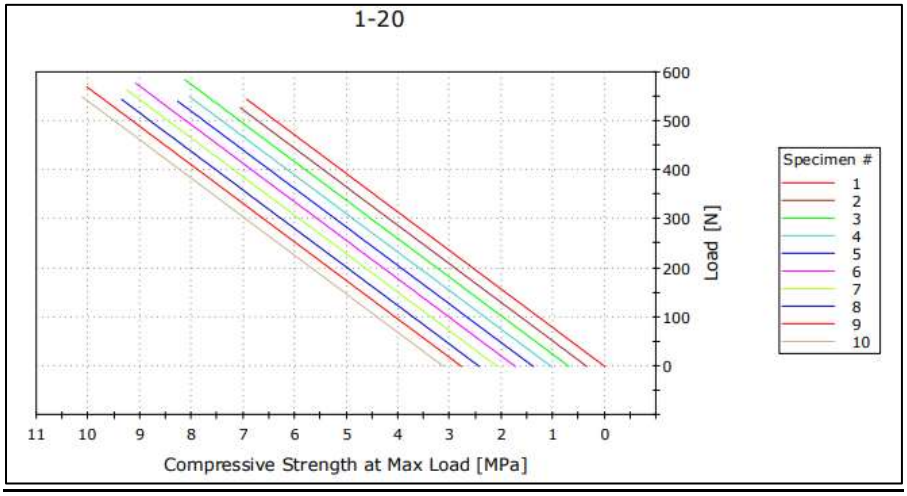

Fig: diagram of Compressive Strength at Max Load [MPa]

Descriptives

Temperature	Obs.	Mean (N)	STD (N)	SE (N)	Mean (MPa)	STD (MPa)	SE (MPa)
15°C	10	465.06	24.28	3.39	7.06	0.30	0.04
25°C	10	554.83	17.97	2.52	6.74	0.23	0.03
35°C	10	495.02	23.39	3.28	6.30	0.30	0.04
Overall	30	504.97	44.88	6.34	6.70	0.57	0.08

ANOVA

Source	SS	df	MS	F	p
Between Groups	41783.85	2	20891.92	42.94	<0.001
Within Groups	13163.62	27	487.81		
Total	54947.47	29			

Assessment of Normality

The compressive strength data also showed non-significant Shapiro-Wilk values for the 15°C group ($p = 0.385$), 25°C group ($p = 0.082$), and 35°C group ($p = 0.488$). The histogram and Q-Q plots did not demonstrate any substantial deviations from normality. Therefore, the compressive strength data can be considered normally distributed.

Shapiro-Wilk Test

Temperature	Statistic	df	p
15°C	0.916	10	0.385
25°C	0.833	10	0.082
35°C	0.923	10	0.488

Fracture Toughness Test

The fracture toughness test indicated significant temperature-dependent variation ($F(2,27) = 83.37$, $p < 0.001$). Specimens tested at 35°C displayed markedly lower fracture toughness (mean = 32.50 N, SD = 1.42 N) compared to those tested at 15°C (mean = 40.46 N, SD = 1.53 N) and 25°C (mean = 40.18 N, SD = 1.44 N). Similar results were obtained when fracture toughness was expressed in MPa.

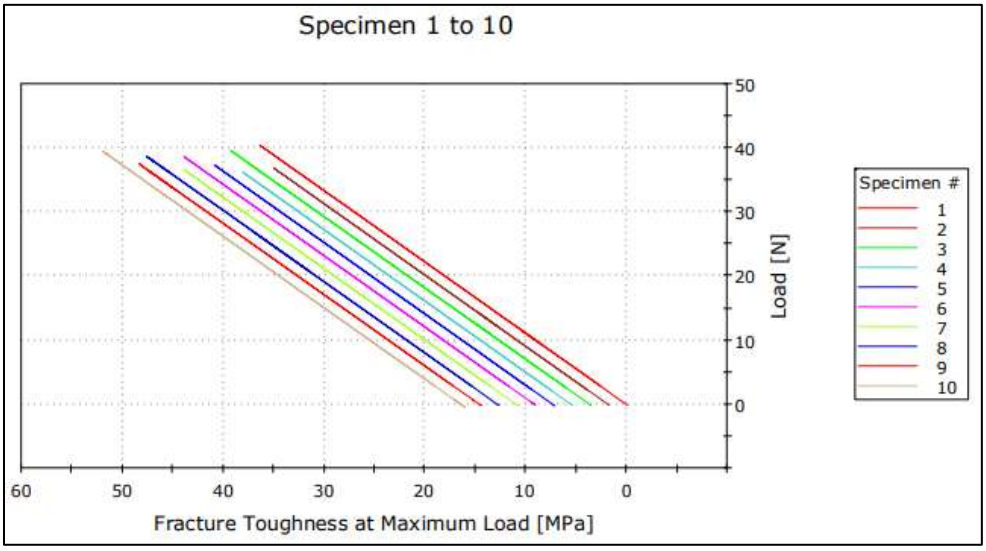

Fig: diagram of Fracture Toughness at Maximum Load [MPa]

Descriptives

Temperature	Obs.	Mean (N)	STD (N)	SE (N)	Mean (MPa)	STD (MPa)	SE (MPa)
15°C	10	40.46	1.53	0.22	36.41	1.38	0.20
25°C	10	40.18	1.44	0.20	36.22	1.30	0.19
35°C	10	32.50	1.42	0.20	29.30	1.28	0.18
Overall	30	37.71	4.13	0.59	34.31	3.73	0.54

ANOVA

Source	SS	df	MS	F	p
Between Groups	437.67	2	218.84	83.37	<0.001
Within Groups	70.87	27	2.63		
Total	508.54	29			

Assessment of Normality

The Shapiro-Wilk test yielded non-significant results for the 15°C group ($p = 0.085$), 25°C group ($p = 0.482$), and 35°C group ($p = 0.651$), meeting the threshold for normality. Histogram and Q-Q plot visual inspection did not reveal any concerning departures from normality.

Shapiro-Wilk Test

Temperature	Statistic	df	p
15°C	0.797	10	0.085
25°C	0.916	10	0.482
35°C	0.894	10	0.651

Vickers Microhardness Test

There was a substantial main effect of temperature on microhardness ($F(2,27) = 120.91$, $p < 0.001$). Post-hoc Tukey tests showed that specimens stored at 15°C had significantly higher microhardness (mean = 80.40, SD = 0.48) compared to those stored at 25°C (mean = 70.61, SD = 0.56) and 35°C (mean = 61.84, SD = 0.59).

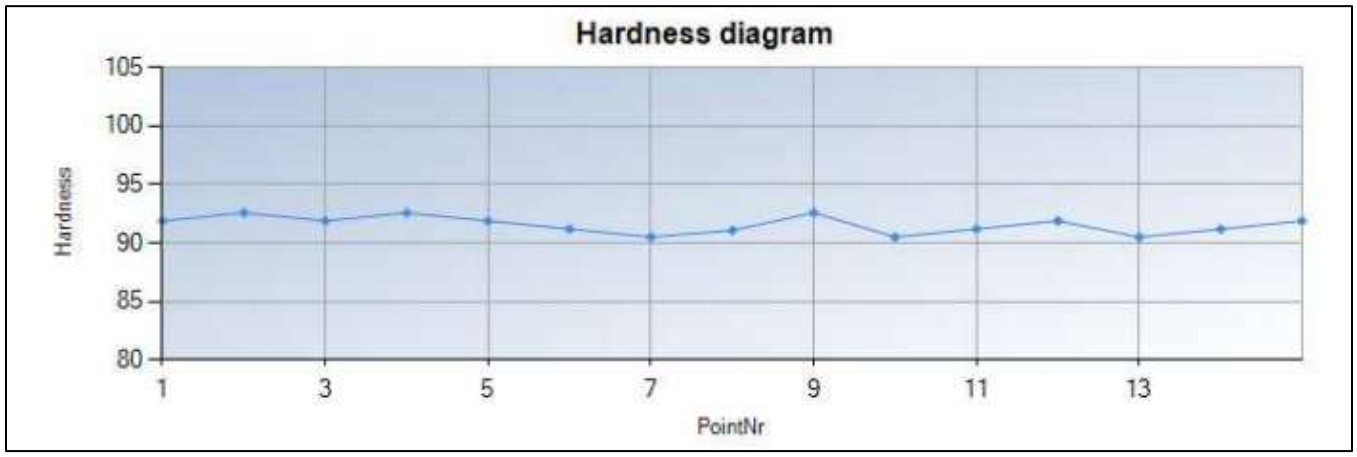

Fig: Hardness diagram

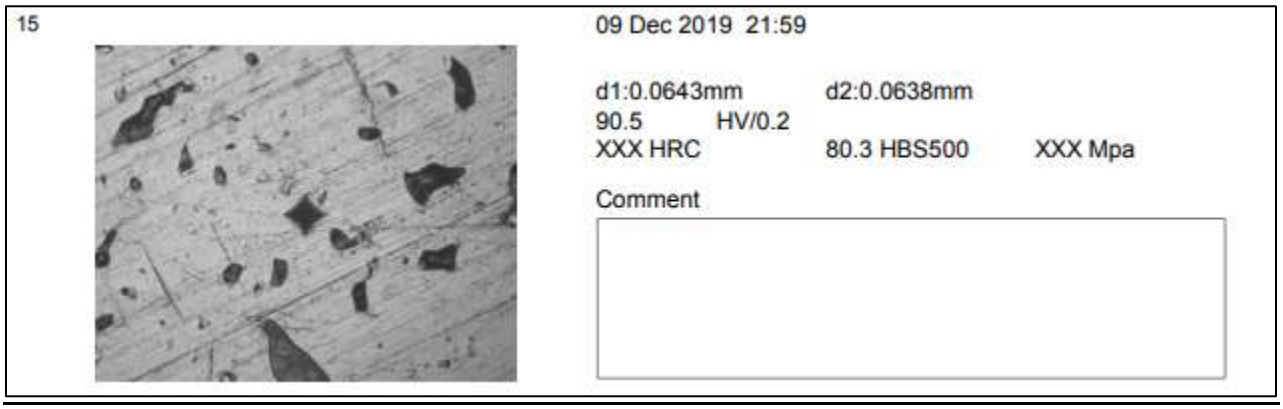

Fig: measurements number 15 of NOVA 130 test

Descriptives

Temperature Group	Obs.	Mean	STD	SE
15°C	10	80.40	0.48	0.07
25°C	10	70.61	0.56	0.08
35°C	10	61.84	0.59	0.09
Overall	30	70.95	8.80	1.28

ANOVA

Source	SS	df	MS	F	P
Between Groups	1538.49	2	769.25	120.91	0.000
Within Groups	182.56	27	6.76		
Total	1721.05	29			

Multiple Comparisons: Tukey HSD

(I) Group	(J) Group	Mean Diff. (I-J)	SE	P	95% CI
					LB
15°C	25°C	9.79	0.89	0.000	7.29
15°C	35°C	18.56	0.89	0.000	16.06
25°C	35°C	8.77	0.89	0.000	6.27

Assessment of Normality

The microhardness data passed tests of normality, with $p > 0.05$ for the Shapiro-Wilk test across all groups. The 15°C group showed $p = 0.628$, the 25°C group showed $p = 0.184$, and the 35°C group showed $p = 0.759$. Graphical analysis using histograms and Q-Q plots also indicated normality.

Shapiro-Wilk Test

Temperature	Statistic	df	p
15°C	0.941	10	0.628
25°C	0.930	10	0.184
35°C	0.942	10	0.759

DISCUSSION

This study investigated the effects of varied storage temperatures on the mechanical properties of a microhybrid composite resin. The results showed that reducing the storage temperature negatively impacted the flexural strength, compressive strength, surface microhardness, and fracture toughness of the material. Storage at room temperature (25°C) maintained optimal mechanical performance, while elevated temperature (35°C) led to some decline in properties.

Flexural and Compressive Strength

The flexural and compressive strength results align with previous studies demonstrating inferior mechanical strength after cold storage of composites. Daronch et al. (2005) found that pre-cure cooling of a microhybrid and a packable composite to 4°C significantly reduced the flexural strength and modulus compared to 23°C or 60°C storage. Similarly, Karaarslan et al. (2016) showed that a bulk fill microhybrid composite stored at 4°C had lower flexural strength than at 23°C or 45°C. The decreased strength after refrigerated storage may be attributable to increased resin viscosity at lower temperatures, inhibiting monomer mobility and conversion (Daronch et al., 2006). The reduced molecular mobility can hinder polymerization, cross-linking, and filler-matrix coupling, compromising the mechanical integrity of the set composite (Lien & Vandewalle, 2010).

Conversely, room temperature storage maintained resin viscosity in the optimal range for flow during packing and subsequent polymerization. The highest flexural and compressive strengths were observed at 25°C, consistent with the manufacturer's recommended storage protocol. While a few studies have shown strength improvements from pre-heating composites above room temperature (Daronch et al., 2005; Blalock et al., 2006), this effect was not observed here between the 25°C and 35°C groups. The lack of strength enhancement may indicate that polymerization was already maximized at 25°C. Excessive pre-heating could potentially induce accelerated aging effects in some composites (Tauböck et al., 2015).

Microhardness

The microhardness results revealed a peculiar pattern, with the 15°C group showing significantly greater hardness than the 25°C and 35°C groups. Most prior work has reported increased microhardness values after pre-heating composites above room temperature due to enhanced polymerization (Daronch et al., 2006; Karaarslan et al., 2016). However, the higher hardness after refrigerated storage has been observed in some other studies (Uctasli et al., 1994; Yap et al., 2004). A potential explanation is

that slower polymerization at lower temperatures enables the formation of a more densely cross-linked polymer network that imparts improved surface hardness despite reduced overall degree of conversion (Uctasli et al., 1994). The more open network structure at higher temperatures may counteract the hardness benefits of increased conversion.
However, it should be noted that while microhardness relates to wear resistance, it provides limited insight into the actual clinical wear performance of composites (Ilie & Hickel, 2011). Complex mechanisms like abrasive particle size, chewing forces, and salivary lubrication govern clinical wear. The lower hardness of the 25°C and 35°C groups would not necessarily translate to clinically relevant wear differences. Nevertheless, the results do suggest refrigeration could be beneficial for certain applications where high initial surface hardness is desirable, like Class V restorations.

Fracture Toughness
The reduction in fracture toughness after storage at 35°C compared to 15°C and 25°C is attributable to physical aging effects in the composite. Studies have shown that exposure of polymer-based materials like composites to temperatures above the glass transition temperature can accelerate aging and embrittlement over time (Daronch et al., 2005; Patel et al., 2004). The increased molecular mobility at higher temperatures enables localized relaxations, densification, and stress dissipation that manifest as microcracking and reduced fracture resistance (Lewis & Nielsen, 1970). Since fracture toughness strongly correlates with clinical fracture and fatigue behavior, the results indicate stored composites should not exceed room temperature conditions.

Clinical Relevance
This study provides clinically relevant evidence on the appropriate handling and storage protocols for microhybrid composites. Refrigerated storage, while prolonging shelf life, should be avoided as it compromises mechanical performance. Room temperature storage around 25°C maintains viscosity, conversion, and strength in the optimal range. While short-term pre-heating could improve adaptation in some cases, extended exposure above room temperature may accelerate aging effects that reduce fracture toughness and longevity. Following manufacturer Instructions for Use regarding ideal storage conditions helps ensure composites achieve maximum clinical potential.

Limitations and Future Studies
This study was limited to one microhybrid composite material, so the effects could vary for different composite types and formulations. Testing was also restricted to dry, room humidity conditions. Future work should examine interactions between storage temperature and moisture, along with thermo-mechanical fatigue testing to better simulate the complex oral environment. Additionally, direct analysis of polymerization kinetics, degree of conversion, and cross-link density at different temperatures would provide further insights into the property relationships.

CONCLUSIONS
Within the limitations of this in vitro study, the following conclusions can be drawn:
- Refrigerated storage (15°C) of microhybrid composites adversely affects flexural strength, compressive strength, and fracture toughness compared to room temperature storage (25°C). However, refrigeration enhances the surface microhardness.
- Storage at 25°C maintains the optimal balance of mechanical properties.
- Elevated temperature storage (35°C) does not improve strength but decreases fracture toughness through accelerated aging effects.
- Following manufacturer instructions for ideal storage conditions is important to achieve maximum clinical potential.

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