

Effect of 1% Nanocurcumin Incorporation on the Flexural Strength, Solubility, and Water Sorption of Calcimol LC and TheraCal PT Pulp Capping Materials: An In Vitro Study

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Abstract

Background and Aim:

Considering the antimicrobial properties of nanocurcumin, this study evaluated the effect of adding 1% nanocurcumin on the flexural strength, water sorption, and solubility of Calcimol LC and TheraCal PT liners.

Materials and Methods:

In this in vitro study, Calcimol LC and TheraCal PT were tested in their original form and with 1 wt% nanocurcumin. Dycal and MTA were used as controls. Flexural strength, water sorption, and solubility were measured according to ISO standards. Data were analyzed using one-way ANOVA and Tamhane's post hoc test. ($\alpha = 0.05$)

Results:

TheraCal PT showed the highest flexural strength, followed by Calcimol LC. The addition of nanocurcumin significantly reduced flexural strength in both materials ($P < 0.001$). MTA and Dycal exhibited the lowest flexural strength. No significant differences were found in water sorption or solubility among groups, and nanocurcumin had no significant effect on these properties. ($P > 0.05$)

Conclusion:

Incorporation of 1% nanocurcumin adversely affected flexural strength but did not significantly influence water sorption or solubility. Although TheraCal PT and Calcimol LC showed superior mechanical properties compared with Dycal and MTA, all materials met acceptable criteria for pulp capping applications.

Introduction

The primary objective of regenerative dentistry is the preservation of pulp vitality through protection of the dental pulp and prevention of its exposure [1]. Direct pulp capping is defined as the application of a protective barrier over the exposed pulp tissue in order to preserve the regenerative potential of pulp cells and promote healing of injured pulp tissue through the use of bioactive agents and induction of mineralized tissue or dentinal bridge formation. Pulp capping agents are also used for indirect pulp capping. Compared with root canal therapy, pulp capping is a more conservative treatment approach [2, 3] and, when performed correctly, demonstrates a success rate ranging from 72.9% to 95.4% [4].

Due to their close contact with the pulp tissue, pulp capping agents should be non-toxic and provide optimal seal for reduction of microleakage. In addition, they should exhibit fluoride release potential, bonding ability to dentin and restorative materials, low solubility and water sorption, optimal

biocompatibility, bioactivity and biointeractivity, dimensional stability, radiopacity, antibacterial properties, high compressive and flexural strength, and a clinically acceptable setting time [5, 6].

Calcium hydroxide [$\text{Ca}(\text{OH})_2$] has been used in dentistry since 1921 because of its antibacterial activity, low cytotoxicity and ability to induce dentinal bridge formation. Given its high clinical success rate, it remained the gold-standard pulp capping agent for many years. [7]. Nevertheless, some disadvantages such as low compressive strength and high solubility limit its application as a base material under restorative materials [8].

Calcimol LC is a light-cured, calcium hydroxide-based liner containing a photoinitiator. It was introduced in 1988 to overcome some of the limitations associated with conventional self-curing calcium hydroxide materials, such as Dycal [9].

Mineral trioxide aggregate (MTA) is another commonly used pulp capping agent that has been shown to produce thicker dentinal bridges with fewer porosities and lower levels of pulpal inflammation compared with calcium hydroxide [10].

TheraCal PT is a resin-modified calcium silicate liner introduced after Biodentine. It is a dual-cure material and has been proposed as an alternative to calcium hydroxide and light-cured resin-modified glass ionomer liners [11].

Gandolfi et al. [6] evaluated the properties of different pulp capping materials and reported that calcium silicate cements, such as MTA Plus gel and MTA Angelus, showed higher porosity, water sorption, and solubility, whereas TheraCal showed lower values.

Nanocurcumin is a bioactive compound with antioxidant, anti-inflammatory, and antimicrobial properties and has very low solubility [12–14]. Previous studies have demonstrated that incorporating curcumin into fissure sealants, can create a coating on the tooth surface and inhibit microbial plaque formation and reduce caries development [15]. Pourhajibagher et al. [15] reported that the addition of 5% nanocurcumin to an ACTIVA Bioactive base/liner produced the largest inhibition zone against *Streptococcus mutans*. Furthermore, Samie et al. [16] showed that nanocurcumin at concentrations of 1%, 2%, and 5% exhibited no cytotoxic effects on pulp cells and promoted the differentiation of undifferentiated dental pulp cells.

However, addition of metallic and non-metallic fillers may alter the compressive or flexural strength of dental materials [17]. Considering the limited information regarding the effect of nanocurcumin incorporation on the mechanical properties of TheraCal PT and Calcimol LC liners, along with the optimal antimicrobial properties of nanocurcumin, this study aimed to evaluate the effect of adding 1% nanocurcumin on the flexural strength, solubility, and water sorption of these materials.

Materials and Methods

Study design and materials

This in vitro experimental study evaluated the mechanical and physicochemical properties of two pulp-capping liners: TheraCal PT (dual-cure calcium silicate liner; Bisco, USA) and Calcimol LC (light-cured calcium hydroxide liner; VOCO, Germany), with and without incorporation of 1 wt% nanocurcumin [18]. Dycal (Dentsply Sirona, USA) and mineral trioxide aggregate (MTA; Angelus, Brazil) were used as control materials. Ethical approval was obtained from the university ethics committee (IR.TUMS.DENTISTRY.REC.1402.044).

Sample size calculation

Sample size was calculated using PASS software (version 11.0; NCSS, LLC, USA) based on previous studies. For water sorption and solubility, assuming a standard deviation of $0.85 \mu\text{g}/\text{mm}^3$, effect size of 0.58, $\alpha = 0.05$, and power of 80% ($\beta = 0.20$), at least eight specimens per group were required. For flexural strength (FS), assuming a standard deviation of 6.5 MPa and an effect size of 0.47 under the same statistical assumptions, at least eleven specimens per group were required [19, 20].

Preparation of nanocurcumin-modified liners

Nanocurcumin powder (Karen Pharmaceutical Co, Yazd, Iran) was incorporated at 1 wt% using a digital balance (accuracy 0.001 g). To ensure homogeneous particle distribution, the powder was first mixed in an empty amalgam capsule containing a metal mixing ball using an amalgamator (Softy, Fakhri Sina Institute, Iran) for 20 s.

The powder was then combined with each liner material in a new capsule and mixed for an additional 20 s. Control specimens were prepared according to manufacturer's instructions using automix syringes. All procedures were performed under controlled ambient lighting to prevent premature polymerization.

TheraCal PT and Calcimol LC specimens were light-cured using an LED curing unit (DTELUX V; Woodpecker, China) with a wavelength of $435 \pm 20 \text{ nm}$ and an intensity of $886 \text{ mW}/\text{cm}^2$. Light intensity was checked after every five curing cycles using a radiometer (Bluephase Meter II; Ivoclar Vivadent, Schaan, Liechtenstein).

Assessment of Water sorption and solubility

Water sorption and solubility were evaluated in accordance with ISO 6876 and ISO 9917-2 standards. Disc-shaped specimens (12 mm diameter $\times$ 2 mm thickness)[21] were prepared using a circular split metal mold placed on a metal base covered with aluminum foil at a controlled temperature of $23 \pm 1^\circ\text{C}$. Table 1 shows the composition of liners used in this study.

Table 1
Composition of liners used in this study

Liner	Composition	Manufacturer
TheraCal PT™	Dual cure resin-modified Calcium silicate Base: silicate glass-mix cement. polyethylene glycol dimethacrylate. bis-GMA. barium zirconate Catalyst: barium zirconate. ytterbium fluoride. Initiator	Bisco. Inc, Schamburg. IL. USA Apply the material in 1mm layers. light cure each layer for 20 s
Calcimol LC	Urethane dimethacrylate resin. calcium dihydroxide. dimethylaminoethyl-methacrylate and TEGDMA	VOCO GmbH. Cuxhaven. Germany
Dycal	Base paste: 1.3-butylene glycol disalicylate. zinc oxide. calcium phosphate. calcium tungstate and iron oxide pigments. Catalyst paste: calcium hydroxide. N-ethyl-o/p-toluene sulfonamide. zinc oxide. titanium oxide. zinc stearate. and iron oxide pigments	Dentsply Tulsa. Dental. Johnson. City. TN. USA
MTA-Angelus	Silicon dioxide or silica (SiO ₂). potassium oxide (K ₂ O). aluminum oxide (Al ₂ O ₃). sodium oxide (Na ₂ O). iron oxide (Fe ₂ O ₃). sulfur trioxide (SO ₃). calcium oxide (CaO). bismuth trioxide (Bi ₂ O ₃). magnesium oxide (MgO). potassium sulphate (K ₂ SO ₄). sodium sulphate (Na ₂ SO ₄). crystalline silica.	QED Ltd.. Bakewell Road. Orton Southgate; Peterborough. Brazil
TEGDMA: triethylene glycol dimethacrylate; bis-GMA: bisphenol A-glycidyl methacrylate		

Materials were manipulated according to manufacturer's instructions. For class 1 (self-cured materials), molds were slightly overfilled and excess material was removed using a 0.5mm thick slide to obtain a flat surface. Specimens were transferred to an incubator at 37 ± 2°C. After 60 minutes from the start of mixing, the samples were removed from the mold and care was taken to avoid surface contamination.

For class 2 (light-cured) and class 3 (dual-cured) materials, a 0.5mm thick slide was glued to the surface, and exposed to light for the recommended time, so that each exposure overlapped the previous one. Finally, the other side of the mold was exposed in the same manner. Immediately after exposure. After curing, specimens were stored at 37 ± 2°C and removed from molds Fifteen minutes after the start of exposure.

All specimen's surfaces were finished using 600-grit silicon carbide abrasive paper (Matador; Hermes Schleifmittel GmbH, Hamburg, Germany). Each specimen was weighed to obtain the initial mass (I) using a precision balance. Specimens were then immersed individually in 20 mL distilled water at 37°C .after 24houres, specimens were removed, surface moisture was gently blotted with absorbent paper,

and the saturated mass (M) was recorded. Subsequently, specimens were dried in an incubator at 37°C until a constant mass was achieved and the final dry mass (D) was recorded.

Water sorption and solubility were calculated ($\mu\text{g}/\text{mm}^3$) using the following equations:

$$\text{Water sorption} = (M - D) / D \times 100$$

$$\text{Solubility} = (I - D) / D \times 100$$

Assessment of flexural strength

Flexural strength was evaluated using the three-point bending test according to ISO 4049. Bar-shaped specimens ($25 \pm 0.1\text{mm} \times 2 \pm 0.1\text{mm} \times 2\text{mm}$) were fabricated using a stainless-steel mold.. The assembly was placed in a leveling press to remove excess material and obtain a smooth specimen surface.

Resin-based liners were placed in two 1mm increments and each layer was light-cured for 20 seconds. Calcium hydroxide and MTA materials were also applied in 2mm thick increments allowed to self-cure according to manufacturer instructions. After setting, all specimens were stored at $37 \pm 2^\circ\text{C}$ until testing.

Each specimen was positioned perpendicular to the supporting rods at the center of the test jig in a universal testing machine. Load was applied at the midpoint at a crosshead speed of $0.75 \pm 0.25\text{mm}/\text{min}$ until fracture occurred (Fig. 1).the applied load for each specimen was recorded using computer software connected to the testing machine and the maximum load at failure (F, Newton) was recorded, and flexural strength (MPa) was calculated using the following equation:

$$\text{flexural strength} = 3FL / 2bh^2$$

Where L is the distance between the supports in millimeters with 0.01mm accuracy, b is specimen width measured at the center of sample before testing (mm), and h is specimen height (mm) measured at the center of sample before testing.

Statistical analysis

Data were analyzed using SPSS software (version 25.0; IBM Corp., USA). Normality and homogeneity of variance were assessed. One-way ANOVA was used for group comparisons. Due to unequal variances, Tamhane's post hoc test was applied. Significance level was set at $\alpha = 0.05$

Results

The mean values and standard deviations of flexural strength, water sorption, and solubility for all study groups are presented in Tables 2–5.

Table 2
Measures of central dispersion for the flexural strength (MPa) of the study groups

Group	Mean	SD	SE	95% CI	
				Lower bound	Upper bound
TheraCal PT	55.52	13.15	4.16	46.11	64.92
TheraCal PT + nanocurcumin	22.09	6.30	1.99	17.58	26.60
Calcimol LC	47.54	13.85	4.38	37.63	57.45
Calcimol LC + nanocurcumin	8.04	6.16	1.95	3.63	12.45
Dycal	5.26	2.29	0.73	3.61	6.90
MTA	1.60	0.95	0.29	0.92	2.28
SD: Standard deviation; SE: Standard error; CI: Confidence interval					

Table 3
Pairwise comparisons of the groups regarding flexural strength (MPa) by the Tamhane's test

Group (I)	Group (J)	Mean difference	SE	P value
TheraCal PT	TheraCal PT + nanocurcumin	33.42	4.61	0.001<
	Calcimol LC	7.98	6.04	0.97
	Calcimol LC + nanocurcumin	47.48	4.59	0.001<
	Dycal	50.26	4.22	0.001<
	MTA	53.92	4.17	0.001<
TheraCal PT + nanocurcumin	Calcimol LC	25.44-	4.81	0.002
	Calcimol LC + nanocurcumin	14.05	2.79	0.001
	Dycal	16.84	2.12	0.001<
	MTA	20.49	2.02	0.001<
Calcimol LC	Calcimol LC + nanocurcumin	39.49	4.79	0.001<
	Dycal	42.28	4.44	0.001<
	MTA	45.94	4.39	0.001<
Calcimol LC + nanocurcumin	Dycal	2.78	2.08	0.97
	MTA	6.44	1.97	0.13
Dycal	MTA	3.66	0.79	0.008
SE: Standard error				

Table 4

Measures of central dispersion for the water sorption ($\mu\text{g}/\text{mm}^3$) of the study groups after 24 hours

Group	Mean	SD	SE	95% CI	
				Lower bound	Upper bound
TheraCal PT	4.24	2.18	0.73	2.56	5.91
TheraCal PT + nanocurcumin	2.78	3.16	1.12	0.14	5.43
Calcimol LC	6.54	2.88	1.02	4.13	8.95
Calcimol LC + nanocurcumin	8.43	5.34	1.89	3.96	12.89
Dycal	8.69	5.49	1.83	4.47	12.90
MTA	15.63	8.96	3.17	8.14	23.11
SD: Standard deviation; SE: Standard error; CI: Confidence interval					

Table 5

Measures of central dispersion for the solubility ($\mu\text{g}/\text{mm}^3$) of the study groups

Group	Mean	SD	SE	95% CI	
				Lower bound	Lower bound
TheraCal PT	5.02	3.73	1.24	2.16	7.89
TheraCal PT + nanocurcumin	4.31	3.86	1.36	1.09	7.54
Calcimol LC	5.64	4.69	1.66	1.71	9.56
Calcimol LC + nanocurcumin	7.79	4.24	1.50	4.24	11.34
Dycal	11.83	7.33	2.44	6.19	17.47
MTA	16.41	10.16	3.59	7.92	24.90
SD: Standard deviation; SE: Standard error; CI: Confidence interval					

Flexural strength

TheraCal PT exhibited the highest flexural strength, whereas MTA showed the lowest value. One-way ANOVA revealed significant differences among the groups ($P < 0.05$). According to Tamhane's post hoc test, pure TheraCal PT demonstrated significantly higher flexural strength than TheraCal PT containing nanocurcumin, Calcimol LC containing nanocurcumin, Dycal, and MTA ($P < 0.001$).

The incorporation of 1% nanocurcumin significantly reduced the flexural strength of both TheraCal PT and Calcimol LC. Calcimol LC also showed significantly higher flexural strength than its nanocurcumin-

containing counterpart, Dycal, and MTA ($P < 0.001$). Dycal exhibited significantly higher flexural strength than MTA ($P = 0.008$). No other significant differences were observed among the groups ($P > 0.05$).

Water sorption

No significant differences were found among the groups regarding water sorption after 24 h ($P > 0.05$). MTA showed the highest mean water sorption value, whereas TheraCal PT containing nanocurcumin exhibited the lowest value.

Solubility

The groups did not differ significantly in solubility values ($P > 0.05$). The highest solubility was observed in MTA, followed by Dycal, while TheraCal PT containing nanocurcumin showed the lowest solubility.

Discussion

The present study evaluated the effect of incorporating 1% nanocurcumin on the flexural strength, water sorption, and solubility of Calcimol LC and TheraCal PT liners. The findings showed that the addition of nanocurcumin significantly reduced the flexural strength of both materials, while no significant changes were observed in water sorption and solubility.

Although differences in solubility among the groups were not statistically significant, MTA and Dycal exhibited higher solubility values than TheraCal PT and Calcimol LC. The relatively high solubility of calcium hydroxide-based materials has been associated with calcium ion release and therapeutic effects [22]. However, excessive solubility may compromise the long-term stability of the material and its sealing ability [23, 24]. MTA may also exhibit increased water uptake due to its hydrophilic nature and intrinsic porosity caused by capillary structure and air entrapment during mixing [25, 26]. Ceci et al. [27], however, reported lower solubility values for MTA, which may be related to differences in storage conditions, evaluation methods, and immersion periods.

Previous studies have reported lower solubility values for resin-modified materials such as TheraCal PT [28, 29]. Since water sorption is generally associated with solubility, materials with lower water sorption often demonstrate lower solubility as well [30]. The lower water sorption and solubility observed in TheraCal PT may be related to its resin-modified calcium silicate structure, which limits moisture diffusion and hydration reactions within the material [31, 32]. Variations among studies may also be attributed to differences in sample dimensions, storage media, and testing periods [33, 34].

In the present study, incorporation of 1% nanocurcumin slightly reduced water sorption in TheraCal PT and slightly increased it in Calcimol LC, although these changes were not statistically significant. This finding may be associated with the small particle size and increased surface area of nanoparticles, which can influence liquid absorption and internal porosity [35]. low concentration of nanoparticles and

their relatively uniform distribution within the material matrix. Reduced nanoparticle agglomeration may improve nanoparticle dispersion within the resin matrix and minimize adverse physicochemical changes [36].

Nevertheless, all tested materials exhibited measurable water sorption, reflecting the inherent hydrophilic nature of dental cements. Porosity and solubility are important factors influencing the durability and stability of dental materials and may be affected by the liquid-to-powder ratio, environmental pH, and material composition [26, 37, 38].

Despite the potential disadvantages of high solubility, increased calcium ion release may enhance antibacterial activity and biological performance [39]. Therefore, a balance between physicochemical stability and ion release is necessary for optimal clinical performance of pulp capping materials. Calcimol LC, as a resin-modified calcium hydroxide liner, demonstrated lower solubility than Dycal and MTA, which may be related to its resin-rich hydrophobic matrix [40–42, 24].

TheraCal PT and Calcimol LC demonstrated higher flexural strength values than Dycal and MTA. The favorable flexural strength of Calcimol LC may be attributed to its resin-rich matrix and hydrophobic monomer composition [9, 43]. In contrast, the lower strength of Dycal has been related to the formation of amorphous calcium disalicylate complexes connected mainly through secondary bonds during polymerization [44, 45]. High water sorption and solubility may further contribute to the lower mechanical properties of Dycal [9].

The incorporation of nanocurcumin significantly reduced the flexural strength of both liners. This reduction may be related to interference of nanoparticles with polymer network formation and incomplete interaction between nanoparticles and the resin matrix [46–48]. Previous studies have similarly reported reductions in mechanical properties following incorporation of curcumin nanoparticles into resin-based materials [49–51]. Although increasing curcumin nanoparticles reduced the flexural strength of acrylic resin, the values remained above the standard limit. [48, 49]. This finding may be attributed to improved nanoparticle dispersion achieved by the mixing method and the low concentration of nanoparticles, which reduced agglomeration within the resin matrix [48].

Comeau et al [50] reported that curcumin incorporation slightly increased water sorption and solubility while minimally affecting flexural strength consistent with the present findings, although not statistically significant. Undissolved nanoparticles may create weak interfaces within the resin matrix, facilitate water diffusion, and reduce the degree of conversion, thereby negatively affecting mechanical performance [50]. Lath et al [51] also reported reduced flexural strength in ACTIVA Bioactive base/liner reinforced with nanocurcumin, especially at higher nanoparticle concentrations.

Flexural strength in the present study was evaluated using the three-point bending test according to ISO 4049 [52]. Differences between the present findings and previous reports on MTA may be attributed to variations in specimen preparation, incubation time, humidity, and mixing procedures [53, 54]. In addition, the physicochemical definition of solubility does not completely correspond to the clinical behavior of

dental cements [55]. Laboratory conditions differ considerably from the oral environment, and the favorable clinical performance of MTA despite its relatively high solubility highlights this discrepancy [56]. Furthermore, the lack of standardized solubility tests for calcium silicate-based materials complicates direct comparisons among studies [57].

This study was conducted under in vitro conditions; therefore, the findings cannot be fully generalized to clinical situations. Factors such as dentin structure, immune responses, oral environment, and long-term functional loading may influence the clinical behavior of pulp capping materials [58]. In addition, only one concentration of nanocurcumin was evaluated. Further studies are recommended to assess different concentrations, other nanoparticles, additional mechanical properties, and the cytotoxicity of nanocurcumin-containing liners.

Conclusion

Within the limitations of this in vitro study, incorporation of 1% nanocurcumin adversely affected the flexural strength of CalciSol LC and TheraCal PT liners, while no significant effect was observed on their water sorption or solubility. Despite the reduction in flexural strength, both materials demonstrated superior physicomaterial properties compared with Dycal and MTA and remained within the acceptable range for use as pulp capping agents. Further studies are recommended to evaluate different concentrations of nanocurcumin, additional mechanical properties, cytotoxicity, and long-term clinical performance.

Declarations

Ethics approval and consent to participate

This study was an in vitro laboratory investigation and did not involve human participants, human tissues, or animals. Nevertheless, the study protocol was reviewed and approved by the Research Ethics Committee of Tehran University of Medical Sciences, School of Dentistry (Approval Code: IR.TUMS.DENTISTRY.REC.1402.044). Therefore, informed consent to participate was not required.

Consent for publication

Not applicable.

Competing interests

The authors declare that they have no competing interests.

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Availability of data and materials

The datasets generated and/or analyzed during the current study are available from the corresponding author upon reasonable request.

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Authors' contributions

Nastaran Dabiri: methodology, investigation, data collection, formal analysis, writing – original draft preparation

Ladan Ranjbar Omrani: Conceptualization, Supervision, study design, methodology, validation, writing – review and editing.

Mohammad javad Kharazifard: Statistical analysis, interpretation of data, visualization.

Fatemesadat Ahmadi: Project administration, resources, supervision, writing – original draft preparation, critical revision of the manuscript, final approval of the manuscript.

All authors read and approved the final version of the manuscript.

Authors' information

Not applicable

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Figures

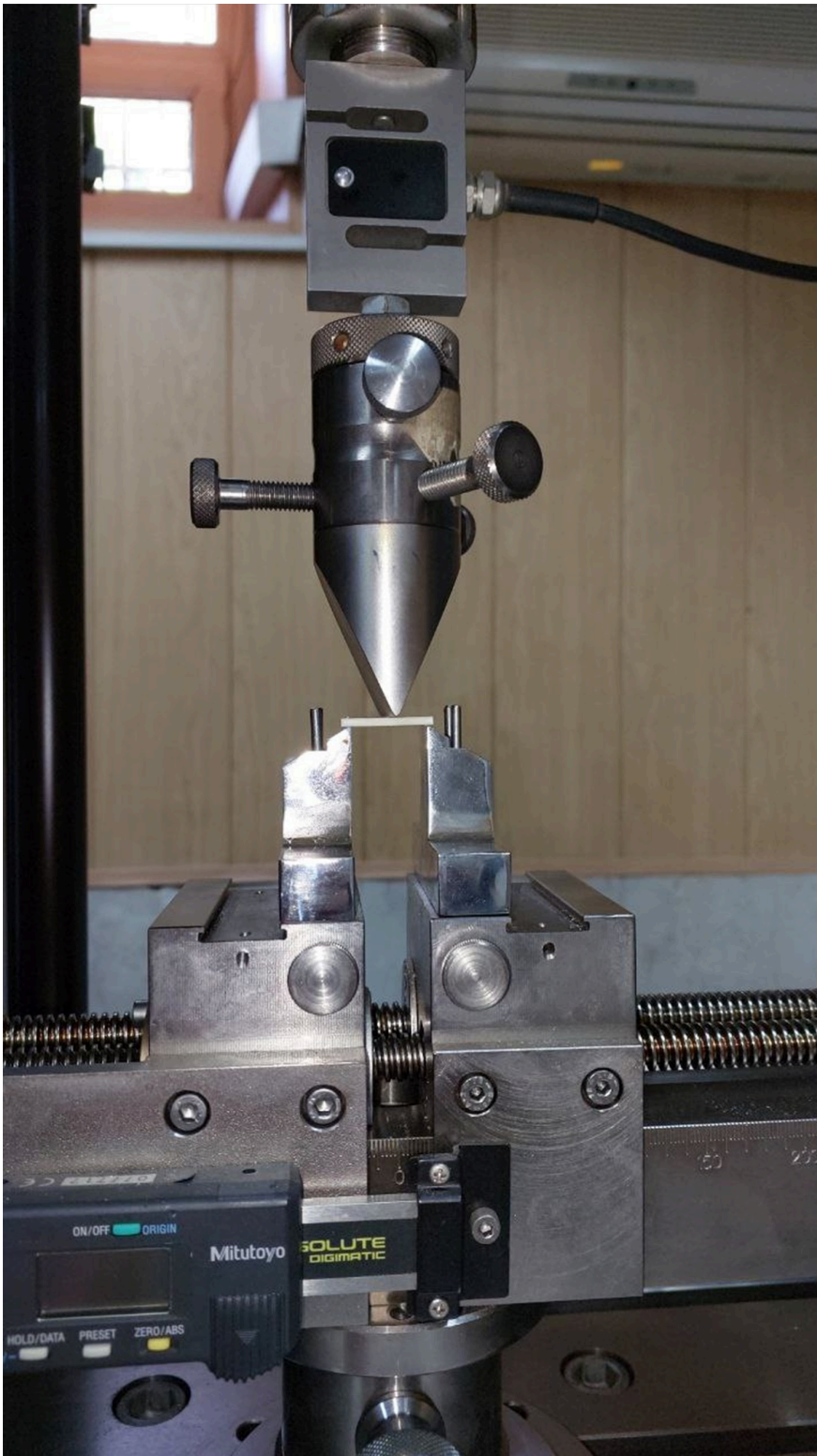


Figure 1

Measuring the flexural strength by the three-point bending test in a universal testing machine.