

## Evaluating the Effect of Nano-Cellulose Fibers Addition on Impact Strength, Transverse Strength and Surface Hardness of Heat-Cured Acrylic Resin Denture Base Material

Maysem Haider Fadhel  \*, Ihab Nabil Safi  

Department of Prosthodontics, College of Dentistry, University of Baghdad, Baghdad, Iraq

### ABSTRACT

The study aimed to find the best amount of cellulose nanofibers to add to heat-cured denture base material to enhance its mechanical characteristics. Cellulose nanofibers (CNF) were added to the polymethyl methacrylate (PMMA) denture base in several weight percentages (0%, 0.5%, 1%, 1.5, and 2%). A probe sonicator was used to mix the monomer with the cellulose nanofibers for around 5 minutes. Impact strength, transverse strength, and shore D surface hardness were the three groups that were classified afterward according to the trials conducted. Descriptive statistics, including means, standard deviations, and bar chart visualisations, were utilised to analyse the data. The findings indicate that the mean values of impact strength and transverse strength measurements exhibited a significant increase in the 0.5% and 1% cellulose nanofiber reinforcement groups, as compared to the control group. However, no significant increase was observed in shore D hardness. Other percentages (1.5% and 2% by weight of CNF) either significantly or insignificantly decreased the mean value of the results. The findings suggest that the incorporation of cellulose nanofibers at concentrations of 0.5% and 1% improves the mechanical properties of a denture foundation.

**Keywords:** Cellulose nanofibers, PMMA, Impact, Transverse strength, and Surface hardness

### 1. INTRODUCTION

Polymethyl methacrylate is the preferred material for the frameworks of dentures. This resin has been widely utilised in the dental field for more than eight decades on account of its low cost, high clinical efficacy, ability to harmonise colours, and stability in intraoral dimensions. Notwithstanding these circumstances, certain challenges persist, such as insufficient surface hardness, diminished strength and fragility, inadequate fatigue and abrasion resistance, and a high incidence of fractures (**Darber et al., 1994**). Fractures are

\*Corresponding author

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one type of mechanical issue that overdenture patients may experience on occasion (**Gibreel et al., 2019**). PMMA, used in denture base materials, has been studied for decades to enhance its mechanical qualities (**Ismail and Muklif, 2015**). (**Gad et al., 2017**) found that micro or nanoscale additives and fibers improve PMMA denture base materials' mechanical qualities. A novel technology reinforces polymers with natural fibers. This makes sense as natural fibers are abundant, renewable, inexpensive, and biodegradable (**Kong et al., 2015**). Ramie or oil palm empty fruit bundle fiber can strengthen a heat-cured PMMA denture foundation (**John et al., 2015; Xu et al., 2013**).

Microscopy improvements have enabled nanotechnology to discover and use nanoparticles to improve the mechanical and physical characteristics of composites, particularly polymer composites. Over the last two decades, several research have examined cellulose nanomaterial, "the future of materials" (**Phuong et al., 2022; Trache, 2020**). Researchers are interested in cellulose nanomaterials because of their biodegradability, abundance in nature, and other important features that enable materials express and enhance function. Due of nano-celluloses' distinctive properties, such as OH groups on their surfaces, nanocomposite polymers have become popular .

Polymer nanocomposites made using nanocelluloses are popular. Cellulose nanofibers have better mechanical qualities, high aspect ratios, and are widely accessible (**Tayeb et al., 2018**). Any cellulose source, including wood fibers, may be used to make cellulose nanofibril or nanofibrillated cellulose using chemical and mechanical procedures (**Jonoobi et al., 2015**). CNF in dental applications greatly improves future success. Its nano-microstructure makes it suitable for mechanical reinforcement (**Cherian et al., 2011**). A few micrometers in length and a diameter on the nanoscale characterize the CNF (**Souza et al., 2015; Souza et al., 2010**). The study's null hypothesis is that the addition 0.5%, 1%, 1.5%, and 2% by wt. Cellulose nanofiber to a PMMA heat-cured denture base material has no meaningful effect. The aim of this study is to select the proper percentage of CNF added to heat-cured PMMA denture base material that improve its mechanical properties.

## 2. MATERIALS AND METHOD

The cellulose nanofibers (CNFs; diameter: 40–80 nm; length: 2-5 $\mu$ m) will be mixed with the acrylic resin monomer in percentages of (0.5%, 1%, 1.5%, and 2%) by weight, with the mixing taking around 5 minutes in the sonicator device. Because of cellulose nanofiber aggregation, percentages above 2% are not used. As soon as the acrylic attained the consistency of dough, packing began. The acrylic resin was removed from the container, rolled, and placed into a mold treated with a separating agent. Both halves of the flask were joined with a polyethylene sheet to ensure the dough spread evenly throughout the mold. A five-minute hydraulic press with a 100 Kp/cm<sup>2</sup> pressure was used. The pressure was relieved, the flask was subsequently unsealed, and the polyethylene sheet was removed. By using a sharp wax knife, all unnecessary material was removed. The second sealing was carried out in the absence of the polyethylene sheet, and the flask was thereafter subjected to pressure (100 Kp/cm<sup>2</sup>) for five minutes. The flask was then securely clamped with the flask clamp to be transferred to a water bath for curing.

The curing process was carried out as per the manufacturer's guidelines. It involved 2 hours of immersing the clamped flask in a water bath and gradually heating it to 70°C. This increase in temperature took 30 minutes. The flask was then kept at this temperature for another 30 minutes. Subsequently, the water was further heated to 100 °C, which took 30 minutes. The flask was maintained at this temperature for another 30 minutes. Following the

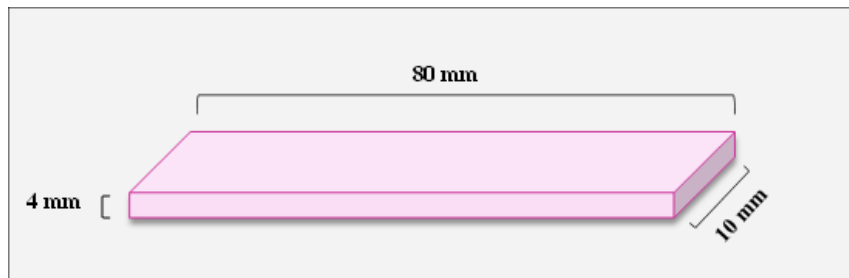
polymerization process, the flasks were allowed to decrease in temperature gradually, and the samples were subsequently placed in distilled water for 48 hours.

## 2.1 Impact Strength Test

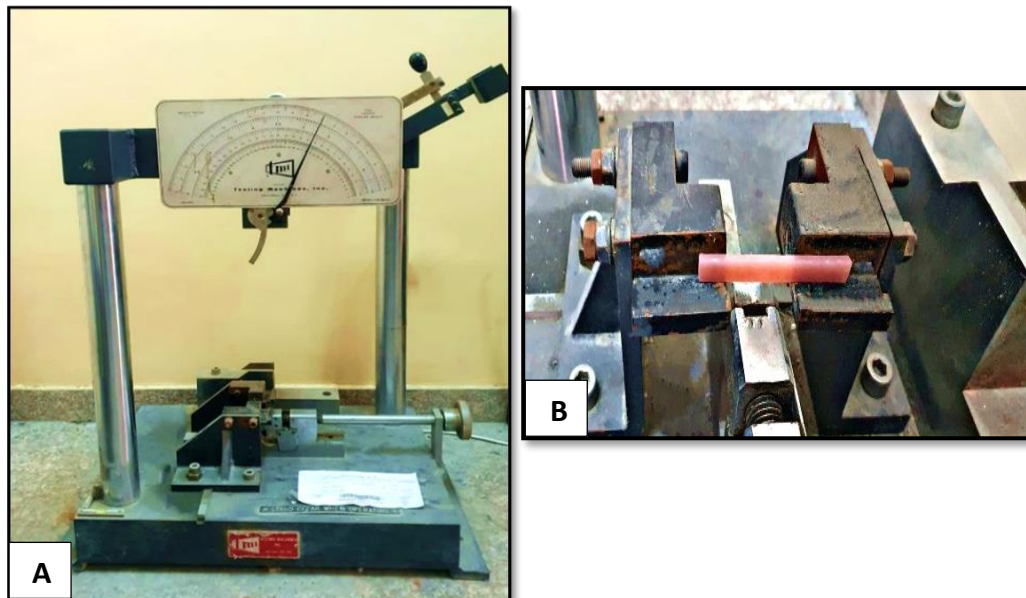
The samples were made with measurements of (80mm x 10 mm x 4 mm) in accordance with **(ISO 179-1, 2000)** as in **Fig.1**, and the test was performed using a Charpy's impact testing apparatus (Testing Machines Inc., USA) in which the specimen was horizontally supported at each end and impacted with a free- rotating pendulum with a 2-joule capacity. A scale records the impact of power absorbed, as in **Fig. 2**. Impact energy in joules per square millimeters, was then calculated using Eq. (1) **(ISO 179-1, 2000)**:

$$\text{Impact strength} = \frac{E}{b.d} \times 10^3 \quad (1)$$

where  $E$  represents the impact power in Joules,  $b$  represents the specimen's width in millimeters, and  $d$  denotes the thickness of the specimen in millimeters. 25 is no. of impact strength specimens



**Figure 1.** The dimensions of the test specimen for impact strength.



**Figure 2.** Impact strength testing A) Impact testing machine; B) Specimen during testing

## 2.2 Transverse Strength

Twenty-five samples 65mm long, 10mm wide, and 2.5 mm thick given by (ADA specification No.12, 1999; Ihab and Moudhaffar, 2011) were manufactured for transverse strength, as in Fig.3. Five samples served as a control group, while the remaining were acrylic specimens to which cellulose nanofibers were added at varying concentrations the number of specimens for each percentage was 5. A universal Instron device was utilized for the examination. Each specimen will be positioned on the testing fixture, which consists of two 50 mm-apart parallel supports. A road positioned in the middle of the supports will apply the stress at a cross-head speed of 1mm/min, leading to deflection until a fracture happens, as in Fig.4. The transverse strength is calculated as:

$$\text{Transverse strength} = \frac{3PI}{2BD^2} \quad (\text{Anusasive et al,2012}) \quad (2)$$

P represents the maximum load, I the span length, B the sample width, and D the sample's thickness.

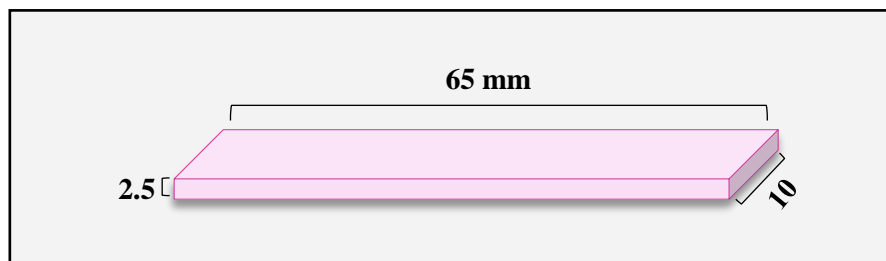


Figure 3. The dimensions of the test specimen for transverse strength.

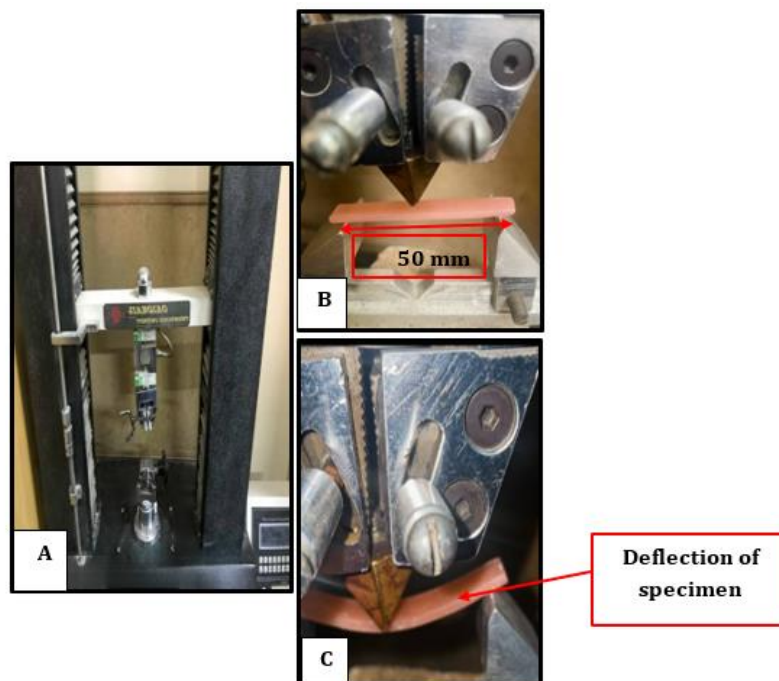
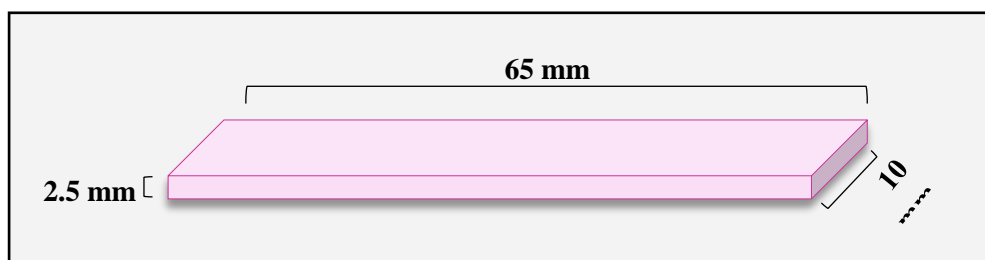


Figure 4. A) Instron testing device; B) Specimen during testing; C) Deflection of specimen.

### 2.3 Shore D Surface Hardness

Twenty-five specimens were fabricated, and the sample utilized in the Shore D hardness measurement should measure (65mm 10mm 2.5mm) as in **Fig.5**. Following the (**ADA Specification No.12, 1999; Ihab and Moudhaffar, 2011**). Five specimens served as a control group. At the same time, the remaining twenty specimens were incorporated with varying concentrations of CNF.

The Shore D durometer hardness tester (Time Group Inc., Italy) was used to measure the surface hardness of the acrylic resin. The device consists of a spring-loaded indenter with a diameter of 0.8mm. An indenter is affixed to a digital scale with a graduated scale ranging from 0 to 100 units. The conventional protocol involves applying swift and forceful pressure on the indenter while documenting the measurement, as in **Fig.6**. Three measurements were made on each specimen, one at the center and one at each extremity. The mean of these measurements was subsequently determined.



**Figure 5.** The dimensions of the test specimen for surface hardness.



**Figure 6.** (A and B) Shore D durometer tester.

### 2.4 Field-emission Scanning Electron Microscopy (FE-SEM)

FE-SEM stands for field-emission scanning electron microscopy. Because of its extreme definition and magnification, FE-SEM is useful for characterizing and developing nano-materials. This enables the observation of tiny details of a sample's surface structure at the nanoscale level, which is important for investigating fine details. One specimen was used for



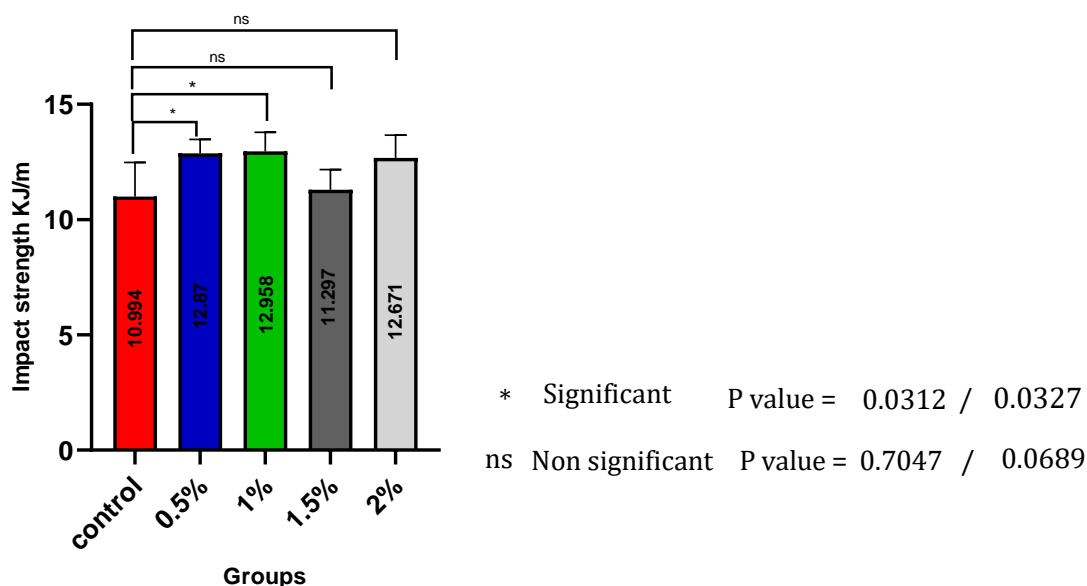
the control group, one for the experimental groups (1% by wt. CNF), and one for CNF powder.

### 3 RESULTS AND DISCUSSION

After addition different percentages of CNF to heat- cured acrylic denture base and 3 tests were analyzed (Impact, Transverse strength and Surface hardness)and examine the specimens under FE-SEM , descriptive statistics including bar chart visualizations and mean values were made by using Graphad Prism program version 9 .

#### 3.1 Impact Strength Test

The impact strength test results following a 48-hour incubation in distilled water showed that impact strength increased in all percentages of addition (0.5, 1, 1.55, 2%) by wt. of CNF. Both experimental groups (0.5% and 1%) had an increased average of mean than the control group and the experimental group (1%) showed the highest mean value of 12.958 KJ/m<sup>2</sup>, as shown in **Fig. 7**. The descriptive statistical analysis and statistical test of impact strength test results using unpaired t-test for comparison of mean values of control and experimental groups are displayed in **Fig. 7**. There was a substantial rise in impact strength in (0.5%, 1%) groups when compared to control and non-significant increase in (1.5% and 2%) groups compared to control.



**Figure 7.** Bar chart for mean values, t-test analysis of Impact strength test.

#### 3.2 Transverse Strength Test

After 48 hours of incubation in distilled water, the transverse strength test results demonstrated an increase in mean values of transverse strength in the CNF addition groups (0.5%, 1%, and 1.5%) in contrast to the control group. However, as indicated in **Fig. 8**, the experimental group (0.5% and 1%) had the highest mean value compared to the control group. Unpaired t-test showed that the addition of (0.5%, 1%) CNF significantly increased



the transverse strength of PMMA (P value < 0.05), while there was a substantial reduction in the group (2%) in contrast to the control group.

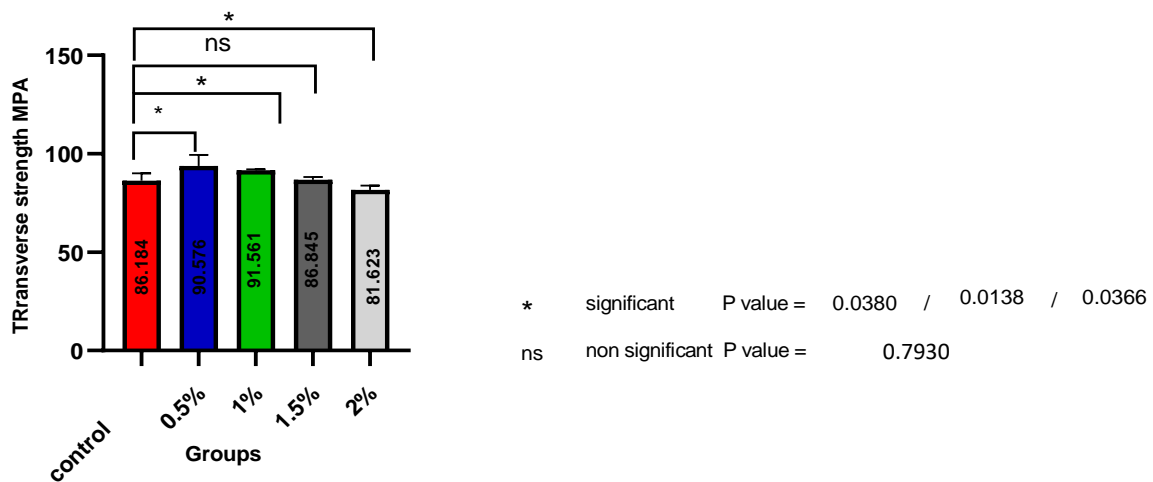


Figure 8. Bar chart of transverse strength test among studied groups.

### 3.3 Shore D Surface Hardness Test

The experimental groups were examined after incubating the acrylic samples in distilled water for 48 hours, and the results demonstrated that adding CNF to PMMA denture base material in percentages (0.5%, 1%, 1.5%) increases the mean surface hardness value. Fig. 9 shows that the 1% group had the greatest mean value among the rest. Statistically, as contrasted with the control group, there is an insignificant increase in shore D surface hardness (P value > 0.05).

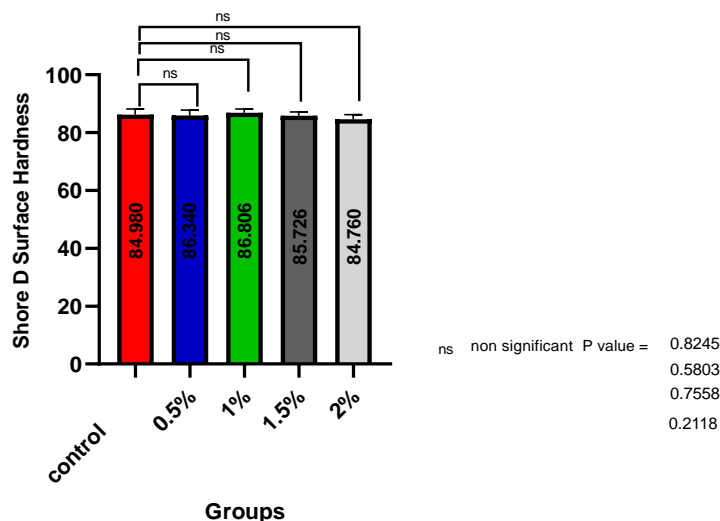
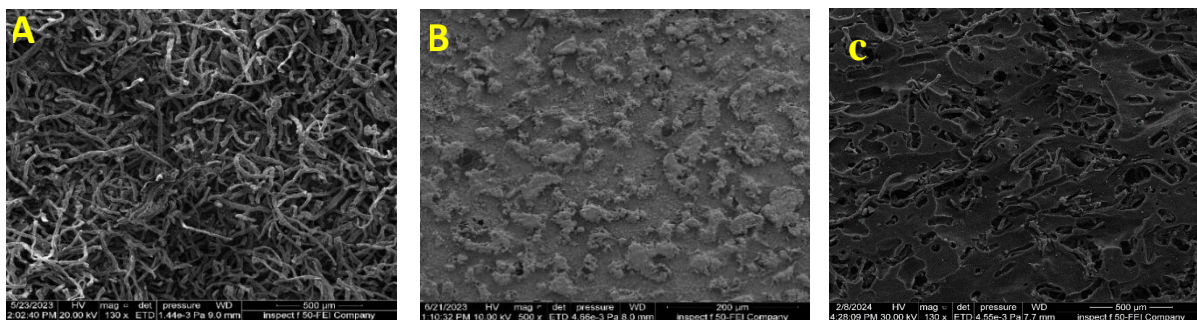


Figure 9. Bar chart for shore D surface hardness p value for each group (0.5, 1, 1.5 , 2%).

### 3.4 Field-emission Scanning Electron Microscopy (FE-SEM)

Fig.10 shows a homogeneous dispersion. of 1% CNF with acrylic resin without agglomeration.



**Figure 10.** FE-SEM micrograph for (A) CNF powder at 130x magnification. ),(b)PMMA heat cured acrylic resin controlled specimens at 500x magnification. (C) PMMA specimens reinforced with 1% CNF at 130x magnification.

The primary disadvantage of natural fibers is their propensity for excessive water absorption, rendering them incompatible with polymer matrices. The reduced interface or adhesion connections between highly water-attracting natural fibers and water-repelling PMMA polymer matrix result in a significant reduction in the characteristics of the composites. CNF treatment with MMA monomer was discovered to be advantageous for forming CNF/PMMA nanocomposite (**Banerjee et al., 2014**). As a result, PMMA-treated CNF disperses better in a polymer of heat-polymerized PMMA. After combining the CNF material with the monomer (methyl methacrylate) of the heat-polymerized acrylic denture base material in the sonicator equipment for 5 minutes, the CNF material was successfully incorporated into the polymethyl methacrylate.

The influence of varied CNF fiber loadings (0.5%, 1%, 1.5%, and 2%) on the transverse, impact, and shore D surface hardness properties was evaluated in this study, during this time the structural and morphological characteristics were examined using FE-SEM analysis. According to the mechanical experiments, introducing CNFs into the PMMA denture base significantly improves impact strength, transverse strength and shore D surface hardness. With their high axial elastic modulus, Large aspect ratio, extensive surface area, and exceptional electrical and thermal characteristics, these fibers can be used as enhancers for polymers, resulting in superior mechanical performance.

However, considerable gains in mechanical characteristics were reported in all experimental tests with the (0.5% and 1%) CNF addition; this may be ascribed to the homogenous distribution of CNFs in the acrylic resin, which is critical to the efficacy of the nanocomposite. Adding nanofibers or nanotubes to PMMA greatly enhanced its properties compared to nanoparticle fillers. The greater surface area to volume ratio of nanotubes and nanofibers compared to nanoparticles is the main reason behind this (a nanofiber's ratio can be up to 103 times that of a microfibrer), and higher mechanical performance, including stiffness and strength when compared to any other form of the material (**Njuguna et al., 2008**). The Nanometric scale can generate massive and extended specific surface areas, up to 1000 m<sup>2</sup>/g (**Njuguna et al., 2007**). Therefore, the stress transfer quality between the matrix material and the nanofibers is improved due to the larger interfacial matrix material surface (interphase) of nanocomposites. This aspect has been identified as a significant determinant in the interface quality of nanocomposites, which facilitates the effective transfer of load from the matrix to the cellulose nanofiber, thus displaying properties quite dissimilar to the bulk polymer (**DE Azeredo et al., 2009**). Another research found that when CNF was added to the epoxy composite increased the impact strength of the epoxy (**Saba et al., 2017**).





Another study discovered that adding a modest amount (0.3 wt.%) of unmodified cellulose nanofibers of pineapple leaves to PMMA nanocomposite enhanced impact strength significantly. They attributed this to the hollow structure of the fiber giving the anti-vibration effect (**Shih et al., 2018**).

The addition of (0.5%, 1%, 1.5%) by wt. CNFs to PMMA increase the mean value of transverse strength in contrast to the control group. This effect is due to CNF's high tensile strength and elastic modulus. These outcomes conform with previous findings for heat-cured acrylic combined with micro-crystalline cellulose fiber extracted from natural oil palm empty fruit bunches (**John et al., 2015**). This conclusion is also consistent with the study of adding CNF to epoxy composites, which increased transverse strength and epoxy modulus (**Saba et al., 2017**). Also, a similar finding was reported in adding cellulose nanofibers to the thermoplastic, injection molded PMMA heat polymerized acrylic resin denture base material (**Kawaguchi et al., 2020**). Another study discovered a significant rise in transverse strength after the addition of 0.5% and 1% by wt. of nanofibers to PMMA denture base material (**Hameed et al., 2022**).

The CNF-reinforced group may depend on the arrangement of fiber particles within the matrix. Augmenting fiber content may reduce flexural strength and enhance nano-filler aggregation. This explains the reduced levels of transverse strength in 2% by wt. of CNF. The addition of (0.5%, 1%, 1.5%) CNF causes an insignificant increase in the mean value of shore D surface hardness of specimens; this may be attributed to the randomly distributed CNF into the acrylic matrix, while in the 2% group, there was a decrease in mean value, this is because of the particle size effect and an increase in the proportion of the fiber, which led to an increase in the cluster in the nanofiller composite., in the nanofiller composite and a decrease in hardness values (**Diya et al., 2018**). PMMA surface hardness was decreased insignificantly with the addition of 1% sisal nanofiber (**Hameed et al., 2022**).

For a long time, scientists have studied ways to increase the interlaminar strength of composites reinforced with fibers, as it is directly related to the composite's dynamic and damage tolerance performance. The issue has been solved using several strategies, including stitching (**Johnson et al., 1983**), Z-pinning (**Marasco et al., 2006**), interleaving (**Chan et al., 1986**), resulting in a substantial increase in toughness and an enhancement of mechanical properties like the lifespan under fatigue conditions. Alternative methods prioritize the adjustment of the interface or matrix characteristics to achieve the desired interlaminar fracture toughness. Crucially, matrix toughening can be achieved using chemical alteration or, more recently, by incorporating fibers and fillers into the matrix material. Moreover, grafting can improve the interfacial and intermolecular compatibility between fibers and matrix (**Xu et al., 2004**). Another study added 0.5% and 1% cellulose nanofibers to the maxillofacial silicon material and found a statistically significant rise in shore A surface hardness of the silicon matrix (**Ali and Safi, 2023**). Hussein reported additional findings when he added zirconia nanoparticles and discovered that surface hardness increased significantly with increasing nano-filler concentration (**Rahman, 2015**). The mean values of the VST50F and Cosmesil M511 elastomers were significantly raised when 0.25 wt.% and 0.2 wt.% TiO<sub>2</sub> nanofillers were added, respectively. (**Shakir and Abdul-Ameer, 2018**).

As shown in **Fig. 10**, FE-SEM revealed a homogenous mixing between CNF and acrylic resin without agglomeration or separation. This is attributed to the well mixing of PMMA monomer and CNF by a probe sonicator. The Young's modulus, tensile strength, and density of a 5 nm-wide bundle of cellulose linear chain molecules are above 130 GPa, 1.7 GPa, and 1.5 g/cm<sup>3</sup>, respectively. Reinforced CNF nanocomposites have low thermal expansion



coefficients, high elastic modulus (around 16 GPa), and elevated tensile strengths (280 MPa) (Iwamoto et al., 2008). Because of these qualities, CNF is an advanced alternative to conventional reinforcing fibers when it comes to dental biomaterials.

#### 4. CONCLUSIONS

Within the scope of this investigation, it is possible to confirm that the addition of cellulose nanofibers to a PMMA heat-cured acrylic denture foundation improves the material's impact strength, transverse strength, and surface hardness. The best results were obtained with CNF concentrations of 0.5% and 1%.

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#### Credit Authorship Contribution Statement.

Maysem Haider Fadhel: Investigation, methodology, software, and Validation  
Ihab N. Safi: Conceptualization, reviewing, and editing.

#### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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## تقييم تأثير إضافة ألياف النانو سليولوز على قوة الصدمة والقوة العرضية والصلابة السطحية لمادة قاعدة طقم الأسنان المصنوعة من راتينج الأكريليك المعالج بالحرارة: دراسة تجريبية

ميسم حيدر فاضل<sup>\*</sup> ، ايهاب نبيل صافي

قسم التعويضات الاصطناعية، كلية طب الأسنان، جامعة بغداد، بغداد، العراق

### الخلاصة

كانت أهداف الدراسة هي العثور على أفضل كمية من ألياف السليولوز النانوية لإضافتها إلى المادة التي يصنع منها قاعدة اطقم الاسنان المعالجة بالحرارة من أجل تعزيز خصائصها الميكانيكية. تمت إضافة ألياف السليولوز النانوية إلى هذه المادة البوليمرية PMMA بعدة نسب وزنية (0% ، 0.5% ، 1% ، 1.5% ، 2%)، مما أدى إلى إنتاج ٧٥ عينة إجمالاً تم تقسيمها على ثلاث مجاميع رئيسية حسب الاختبارات الثلاث، قوة الصدمة والقوة العرضية وصلابة السطح . بعد ذلك تقسيم كل مجموعة الى خمس مجاميع متساوية العدد حسب التراكيز المضافة من المادة النانوية ، واحدة بدون CNF، وواحدة بنسبة 0.5% ، وواحدة بنسبة 1.5% ، ومجموعتين بنسبة 2% من الوزن. علاوة على ذلك تم تحليل المقطع العرضي للعينات باستخدام المجهر الالكتروني الماسح عالي الدقة. تم استخدام الإحصائيات الوصفية، بما في ذلك تصورات المخطط الشريطي، والانحراف المعياري، والمتوسط، لتحليل البيانات. بينت النتائج انه بالمقارنة مع مجموعة الاكريليك من غير اضافة المادة النانوية ، فإن متوسط قيمة قوة الصدمة، واختبارات القوة العرضية لمجموعتي تقوية ألياف السليولوز النانوية 0.5% و 1% زادت بشكل ملحوظ، في حين أن الزيادة في صلابة السطح لم تكن كبيرة جداً. النسب الأخرى المضافة من المادة النانوية (1.5% ، 2%) أدت إلى انخفاض متوسط قيمة النتائج. تم الاستنتاج انه بإضافة 0.5% و 1% من مادة الياف السليولوز النانوية حسنت و بشكل ملحوظ الخواص الميكانيكية للمادة الاساسية لاطقم الاسنان المتحركة

**الكلمات المفتاحية:** قوة الصدمة ، القوة العرضية ، صلابة السطح ، الياف السليولوز النانوية