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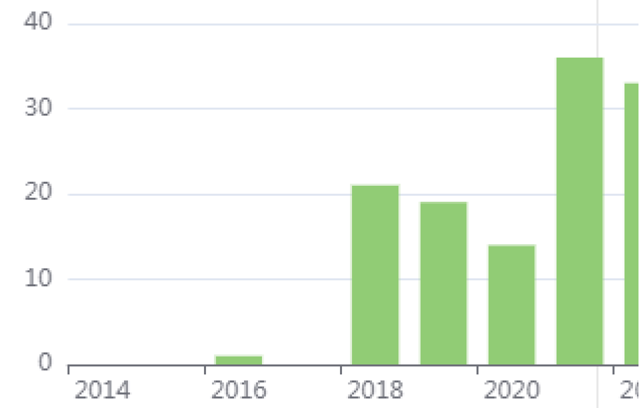


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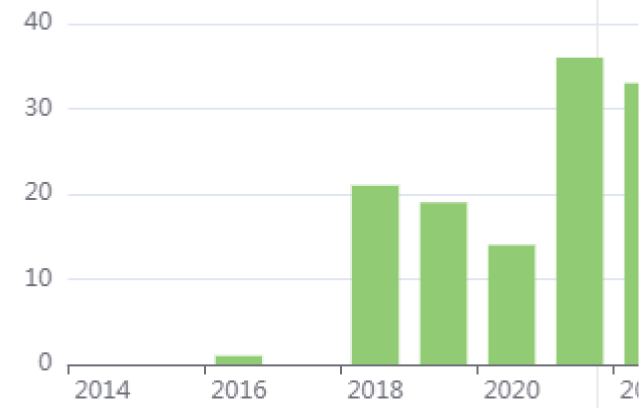


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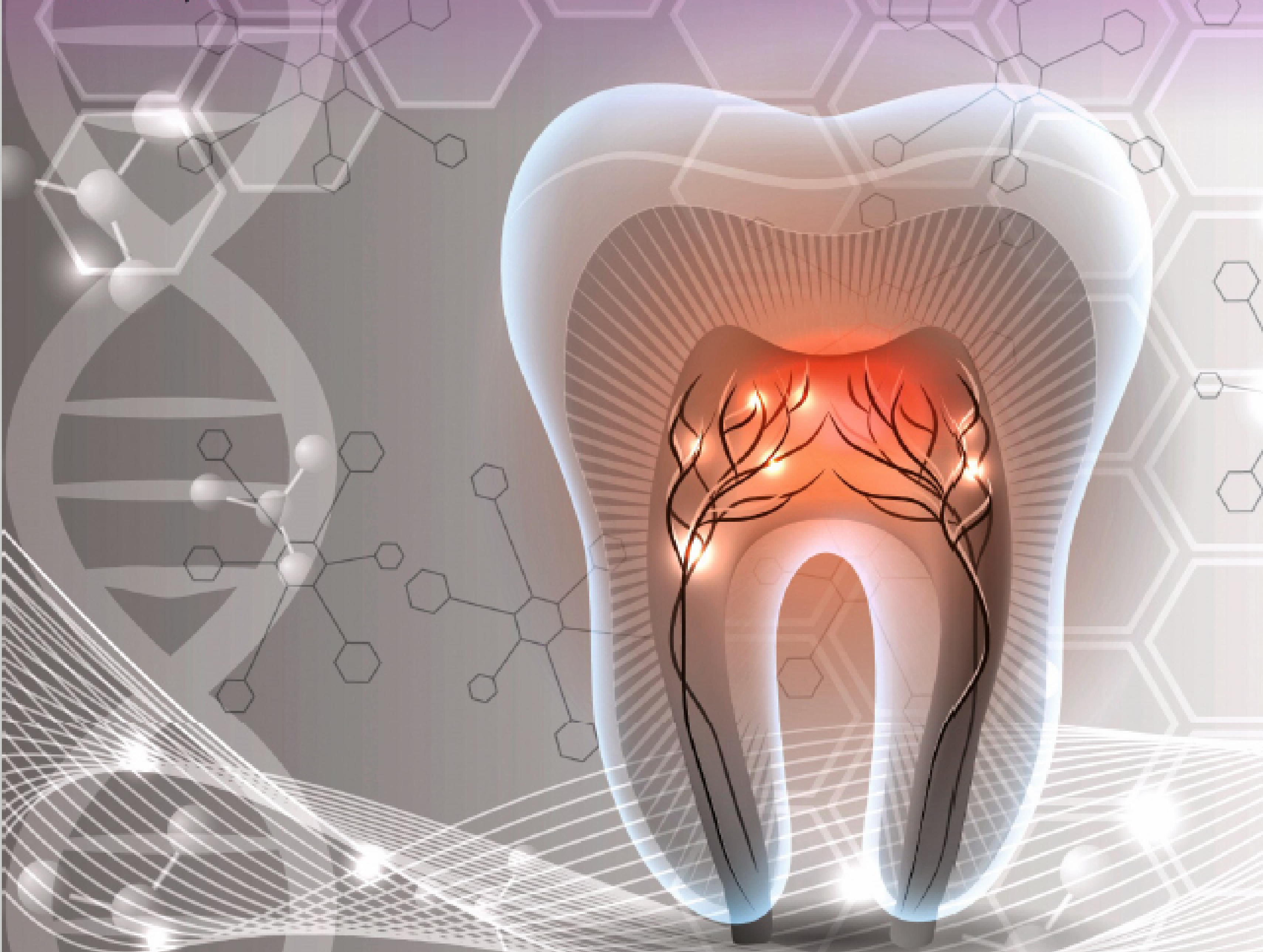

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## ORIGINAL ARTICLES

Original Article

# The Effects of Reinforced Cellulose Nanocrystals from Sugarcane Bagasse Fiber on the Hardness of Glass Ionomer Cements

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**Abstract**

**Background:** Advances in nanotechnology research make the use of cellulose nanocrystals (CNCs) attractive for improving the mechanical properties of glass ionomer cement (GIC). Sugarcane bagasse (*Saccharum officinarum* L.) is a CNCs source with a high CNC content (72.5%). **Objective:** This study aimed to determine the effect of the addition of sugarcane bagasse CNCs on the mechanical properties of GIC. **Methods:** In total, 42 GIC (Fuji IX, GC, Japan) samples were divided into six groups, with various concentrations of CNCs, added to the samples. After 24 h immersion in distilled water at 37°C, the samples were analyzed using the Vickers hardness test. The samples were also characterized by transmission electron microscopy (TEM). For statistical analysis, a one-way analysis of variance, followed by Tukey's *post hoc* test, was applied. A value of $P < 0.05$ denoted statistical significance. **Results:** The TEM revealed crystalline particles in the form of nanocrystals, with varying particle sizes (lengths of 100–200 nm and diameters of 4–19 nm). The addition of 0.4% of CNCS from bagasse fiber to GIC increased the Vickers hardness of the material by 38.89% ($P < 0.05$). **Conclusion:** The addition of 0.4% of sugarcane bagasse can improve the hardness of GIC.

**Keywords:** *Bagasse, cellulose nanocrystal, glass ionomer cement, sugarcane, Vickers hardness*

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## Introduction

Glass ionomer cement (GIC) is a restorative material widely used in dental clinics because of its ability to adhere to enamel and dentine, which in turn, releases fluoride. GIC has poor mechanical properties, which limits its use in high-stress areas.[1-3] To improve the mechanical properties of GIC, several modification has been done, in particular, the addition of different amounts of cellulose nanocrystals (CNC)s from eucalyptus wood.[4] The study led by Silva *et al.* found that 50% CNCs from eucalyptus wood added into GIC marked improvements in various mechanical properties (e.g., compressive strength, elastic modulus, and diametral tensile strength), but there is no information regarding the Vickers hardness value.[4]

Cellulose-based nanomaterials have the potential for biocomposites development in industrial and biomedical applications.[5] Cellulose is an abundant biopolymer in nature, biodegradable and nontoxic, with a low density and good mechanical properties.[6] Cellulose is found in plant cell walls in wood, cotton, hemp, and other plant-based materials and plays an essential role in plant structure.[7]

Sugarcane (*Saccharum officinarum L.*) bagasse is a potential source of cellulose in the production of crystalline nanocellulose.[8] According to the literature, around 640–660 Mton of sugarcane can produce 160 Mton of bagasse. As shown in previous research, the crystallinity value of CNCs from bagasse was higher (72.5%) than that of chemically purified

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cellulose (63.5%).[7] Studies also demonstrated that the crystalline phase was essential in increasing the strength of the material.[9] Nevertheless, no further information regarding the efficacy of sugarcane CNCs' augmentation into GIC. Considering this research gap, this study aims to determine the effect of sugarcane CNCs' addition on the hardness of GIC.

## Materials and Methods

CNCs from sugarcane bagasse was prepared using the basic hydrolysis method, followed by bleaching and acidic hydrolysis. Basic hydrolysis was carried out by dissolving 30 g of dried sugarcane bagasse in sodium hydroxide 4M. As the hydrolysis product was in the form of a suspension, filtration of a suspension was first performed. The filtrate was then compressed. Subsequently, the bagasse was bleached using 1.25% sodium hypochlorite solution. The dried cellulose was added to 45% sulfuric acid solution. Following ultrasonication for 10 min, the dried cellulose was centrifuged at a speed of 10,000 rpm for 10 min and then filtered using filter paper to obtain CNCs in gel form. The synthesized CNCs were then characterized using transmission electron microscopy (TEM) (Hitachi HT7700, Tokyo, Japan) with ×30,000. The CNCs lengths and diameters were measured. In this study, TEM was used because it has a focus of light, which contains high electron energy so that it can analyze the microstructural of a specimen, the crystal structure with high resolution compared to scanning electron microscopy, which can only penetrate the surface of the sample.

The CNCs was then weighed and added to a GIC matrix of a conventional GIC (Fuji IX, GC, Tokyo, Japan, LOT 1804051). There were six experimental groups, whereas each group consists of seven GIC's. Each group was incorporated with different concentrations of CNCs: GIC-CNCs 1%; GIC-CNCs 0.8%; GIC-CNCs 0.6%; GIC-CNCs 0.4%, and GIC-CNCs 0.2%, along with a control group of GIC without CNCs, respectively. These concentrations are based on the research conducted by Silva *et al*. about the addition of CNCs from eucalyptus wood to GIC.[4] After the addition of the various concentrations of CNCs to the samples, the mixture was sonicated for 2 min to produce a GIC powder, with a powder/liquid ratio of 1/1. Materials for GIC were then manipulated manually according to the manufacturer's recommendations. Samples of GIC-CNCs were produced with 3-mm thick and 5 mm in diameter. Before testing the mechanical properties of the samples using the Vickers hardness test, the samples were immersed in distilled water for 24 h at 37°C (±1°C).[10]

### Statistical analysis

In each group, the hardness values were calculated, with the average value and standard deviation recorded. To analyze the average value of each test group, the Ryan–Joiner normality test was used. As all the data were normally distributed, a one-way analysis of variance (ANOVA) statistical test was performed, followed by Tukey's *post hoc* test. A value of $P < 0.05$ denoted statistical significance.

## Results

The TEM characterization of the morphology of the crystalline particles in the CNCs revealed whiskers (crystallinity index: 75%) with varying particle sizes (lengths of 100–200 nm and diameters of 4–19 nm). which is shown in Figure 1.

The mean values and standard deviations in the Vickers hardness test are summarized in Table 1. The statistical analysis revealed a significant difference in the Vickers hardness among the groups ($P < 0.05$) and pointed to a substantial increase in the Vickers hardness of the GIC in all the groups. The maximum Vickers hardness was obtained at CNCs concentration of 0.4%.

Table 2 shows the results of the statistical analysis of the differences in the Vickers hardness using a one-way ANOVA and the Games–Howell *post hoc* test. As shown in the table, there were significant differences in the hardness values of all the groups ($P < 0.05$), with the exception of the GIC-CNCs 0.2% versus the GIC-CNCs 0.6% group ($P = 0.960$), GIC-CNCs 0.2% versus the GIC-CNCs 0.8% group ($P = 0.996$), GIC-CNCs 0.2% versus the GIC-CNCs 1%group ($P = 0.928$), GIC-CNCs 0.6% versus the GIC-CNCs 0.8% group ($P = 0.999$),

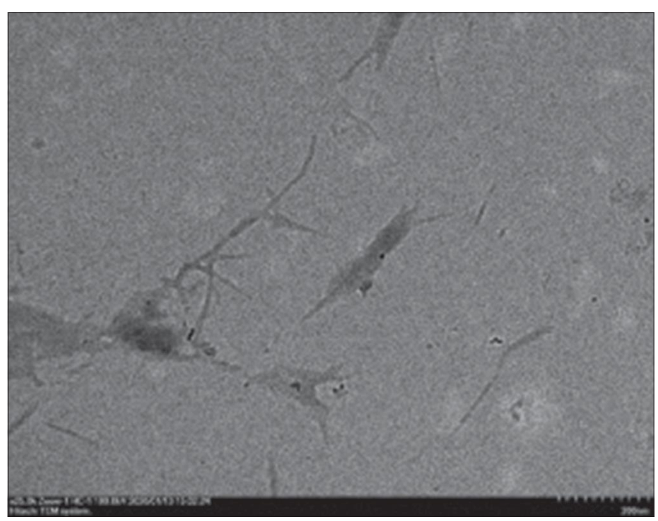

**Figure 1:** Transmission electron microscopy images showing cellulose nanocrystals whisker-shaped particles at × 30,000

GIC-CNCs 0.6% versus the GIC-CNCs 1% group ($P$ = 0.466), and the GIC-CNCs 0.8% versus the GIC-CNCs 1% ($P$ = 0.689) group.

## Discussion

GIC has poor mechanical properties, such as low tensile strength and compressive strength compared to other restorative materials, such as composite resin.[11,12] Surface hardness is an important factor in controlling wear resistance and thus can be used as an indication of the long-term durability of materials.[13] The reduced surface hardness of dental restoration leads to a decrease in wear resistance. Recent studies showed that the microhardness of GIC served as a valid measure of the surface mechanical properties of the material.[14]

Advances in nanotechnology research have made the use of CNCs attractive for improving the mechanical properties of GIC.[14] Nanotechnology, also known as molecular nanotechnology or molecular engineering, has been introduced in the dental field, with GIC containing 3 and 5% titanium dioxide nanoparticle showed improved fracture toughness.[14] The nanoscale particles and crystals in CNCs have similarities to their crystals (nanomaterial) found in natural teeth. This study used CNCs synthesized from sugarcane bagasse and visualized the microstructure of the particles at the nanoscale using TEM. The results of the TEM analysis revealed whisker-shaped nanoparticles in the form of separate aggregates. The average lengths and diameters of the CNCs were 100–200 nm and 4–10 nm, respectively. Cellulose nanowhiskers are rod-shaped particles, which have high particle crystallinity, a crystallinity index >75%, and rectangular cross-sections with few defects.[15-17] As a result, the material containing cellulose nanowhiskers is stronger than metal.

In this study, various concentrations of CNCs were added to GIC samples. The right concentration can provide the ideal interaction between the crystal and cement matrix during a chemical reaction with the reinforcing structure formation.[12] As demonstrated previously, the concentration and intrinsic character of the reinforcing agent added to GIC affected the hardness of the resulting matrix.[4] In this study, the addition of CNCs as a reinforcing agent significantly increased the Vickers hardness value of the tested samples.

It is important to analyze the hardness of dental materials to ensure their clinical function, including resistance to masticatory forces.[18] Hardness testing has frequently been used to evaluate the surface resistance of materials to plastic deformation caused by penetration.[18] Hardness was shown to be closely related to compressive, flexural, and wear properties.[19] In the present study, hardness testing revealed the highest hardness value (38.89%) in the GIC-CNCs 0.4% group, at concentrations of CNCs above 0.4%, more glass particles were found on the surface so the hardness value decreases. The same thing was also found in a previous study, which showed decreased in Vickers micro-hardness from GIC reinforced with CNC from eucalyptus wood due to the large number of glass particles on the surface of the GIC.[4] As demonstrated previously, the proportion of glass particles and polyacid affected the hardness of GIC, its proportion influenced the concentration of filler required to produce a material with a good hardness value.[19] A previous study showed that fewer glass particles on the surface of GIC resulted in a low glass to polyacid ratio, which led to an increased ability of polyacids reaction with the nanoparticles.[20] In the same study, interstitial packing of these nanoparticles resulted in a higher nanoparticle: Matrix ratio at the interface, where the size constraints of the larger glass particles contributed to a surface layer rich

**Table 1: The Vickers hardness test results**

| Groups | Vickers hardness | |
|---|---|---|
| | Mean±SD (VHN) | Means (%)* |
| GIC | 103.96±8.73 | 0 |
| GIC-CNCs 0.2% | 126.01±6.97** | 18.32 |
| GIC-CNCs 0.4% | 144.40±8.29** | 38.89 |
| GIC-CNCs 0.6% | 128.02±6.50** | 23.14 |
| GIC-CNCs 0.8% | 127.20±9.47** | 22.35 |
| GIC-CNCs 1% | 123.70±9.44** | 18.99 |

*The percentage comparison means value between each group and the control group, **$P$<0.05: statistically significant difference compared with control group (GIC). VHN: Vickers hardness numbers, SD: Standard deviation, GIC: Glass ionomer cement, CNCs: Cellulose nanocrystals

**Table 2: Statistical analysis of the differences in the Vickers hardness means among the six groups using a one-way analysis of variance and the Games-Howell *post hoc* test**

| Groups | GIC | GIC-CNCs 0.2% | GIC-CNCs 0.4% | GIC-CNCs 0.6% | GIC-CNCs 0.8% | GIC-CNCs 1% |
|---|---|---|---|---|---|---|
| GIC | - | 0.000* | 0.000* | 0.000* | 0.000* | 0.000* |
| GIC-CNCs 0.2% | - | - | 0.000* | 0.960 | 0.996 | 0.928 |
| GIC-CNCs 0.4% | - | - | - | 0.000* | 0.000* | 0.002* |
| GIC-CNCs 0.6% | - | - | - | - | 0.999 | 0.466 |
| GIC-CNCS 0.8% | - | - | - | - | - | 0.689 |
| GIC-CNCs 1% | - | - | - | - | - | - |

*$P$<0.05: Statistically significant difference. GIC: Glass ionomer cement, CNCs: Cellulose nanocrystals

in the matrix. Furthermore, larger glass particles size led to the more surface layer fill with matrix.[19]

The same study showed the integrity of the interface between glass particles and the matrix. In the present study, the increase in the micro-hardness value of the GIC-CNCs 0.4% group indicates the interaction of the filler and the matrix, which results in an ideal proportion of glass particles and acids on the GIC surface to reacts with the nanoparticles.

## Conclusion

The addition of CNCs from sugarcane bagasse significantly increased the Vickers hardness of GIC restorative material. Further studies are needed to investigate the bonding ability of GIC-containing CNCs from sugarcane bagasse with the tooth structure, its fluoride release ability, and biocompatibility, including clinical trials to further understand the properties and characteristics of this material, especially in the oral environment.

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Nil.

### Conflicts of interest

There are no conflicts of interest.

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# The Effects of Reinforced Cellulose Nanocrystals from Sugarcane Bagasse Fiber on the Hardness of Glass Ionomer Cements

*by* Joko Kusnoto

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Original Article

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**Abstract**

**Background:** Advances in nanotechnology research make the use of cellulose nanocrystals (CNCs) attractive for improving the mechanical properties of glass ionomer cement (GIC). Sugarcane bagasse (*Saccharum officinarum* L.) is a CNCs source with a high CNC content (72.5%). **Objective:** This study aimed to determine the effect of the addition of sugarcane bagasse CNCs on the mechanical properties of GIC. **Methods:** In total, 42 GIC (Fuji IX, GC, Japan) samples were divided into six groups, with various concentrations of CNCs, added to the samples. After 24 h immersion in distilled water at 37°C, the samples were analyzed using the Vickers hardness test. The samples were also characterized by transmission electron microscopy (TEM). For statistical analysis, a one-way analysis of variance, followed by Tukey's *post hoc* test, was applied. A value of $P < 0.05$ denoted statistical significance. **Results:** The TEM revealed crystalline particles in the form of nanocrystals, with varying particle sizes (lengths of 100–200 nm and diameters of 4–19 nm). The addition of 0.4% of CNCS from bagasse fiber to GIC increased the Vickers hardness of the material by 38.89% ($P < 0.05$). **Conclusion:** The addition of 0.4% of sugarcane bagasse can improve the hardness of GIC.

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## Results

The TEM characterization of the morphology of the crystalline particles in the CNCs revealed whiskers (crystallinity index: 75%) with varying particle sizes (lengths of 100–200 nm and diameters of 4–19 nm). which is shown in Figure 1.

The mean values and standard deviations in the Vickers hardness test are summarized in Table 1. The statistical analysis revealed a significant difference in the Vickers hardness among the groups ($P < 0.05$) and pointed to a substantial increase in the Vickers hardness of the GIC in all the groups. The maximum Vickers hardness was obtained at CNCs concentration of 0.4%.

Table 2 shows the results of the statistical analysis of the differences in the Vickers hardness using a one-way ANOVA and the Games–Howell *post hoc* test. As shown in the table, there were significant differences in the hardness values of all the groups ($P < 0.05$), with the exception of the GIC-CNCs 0.2% versus the GIC-CNCs 0.6% group ($P = 0.960$), GIC-CNCs 0.2% versus the GIC-CNCs 0.8% group ($P = 0.996$), GIC-CNCs 0.2% versus the GIC-CNCs 1%group ($P = 0.928$), GIC-CNCs 0.6% versus the GIC-CNCs 0.8% group ($P = 0.999$),

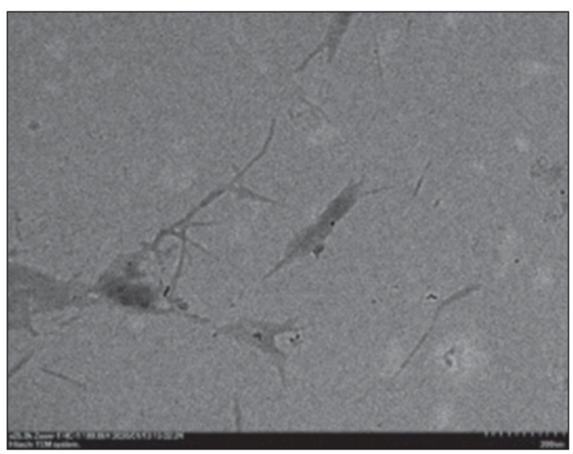

**Figure 1:** Transmission electron microscopy images showing cellulose nanocrystals whisker-shaped particles at × 30,000

GIC-CNCs 0.6% versus the GIC-CNCs 1% group (*P* = 0.466), and the GIC-CNCs 0.8% versus the GIC-CNCs 1% (*P* = 0.689) group.

## Discussion

GIC has poor mechanical properties, such as low tensile strength and compressive strength compared to other restorative materials, such as composite resin.[11,12] Surface hardness is an important factor in controlling wear resistance and thus can be used as an indication of the long-term durability of materials.[13] The reduced surface hardness of dental restoration leads to a decrease in wear resistance. Recent studies showed that the microhardness of GIC served as a valid measure of the surface mechanical properties of the material.[14]

Advances in nanotechnology research have made the use of CNCs attractive for improving the mechanical properties of GIC.[14] Nanotechnology, also known as molecular nanotechnology or molecular engineering, has been introduced in the dental field, with GIC containing 3 and 5% titanium dioxide nanoparticle showed improved fracture toughness.[14] The nanoscale particles and crystals in CNCs have similarities to their crystals (nanomaterial) found in natural teeth. This study used CNCs synthesized from sugarcane bagasse and visualized the microstructure of the particles at the nanoscale using TEM. The results of the TEM analysis revealed whisker-shaped nanoparticles in the form of separate aggregates. The average lengths and diameters of the CNCs were 100–200 nm and 4–10 nm, respectively. Cellulose nanowhiskers are rod-shaped particles, which have high particle crystallinity, a crystallinity index >75%, and rectangular cross-sections with few defects.[15-17] As a result, the material containing cellulose nanowhiskers is stronger than metal.

**Table 1: The Vickers hardness test results**

| Groups | Vickers hardness | |
|---|---|---|
| | Mean±SD (VHN) | Means (%)* |
| GIC | 103.96±8.73 | 0 |
| GIC-CNCs 0.2% | 126.01±6.97** | 18.32 |
| GIC-CNCs 0.4% | 144.40±8.29** | 38.89 |
| GIC-CNCs 0.6% | 128.02±6.50** | 23.14 |
| GIC-CNCs 0.8% | 127.20±9.47** | 22.35 |
| GIC-CNCs 1% | 123.70±9.44** | 18.99 |

*The percentage comparison means value between each group and the control group, ***P*<0.05: statistically significant difference compared with control group (GIC). VHN: Vickers hardness numbers, SD: Standard deviation, GIC: Glass ionomer cement, CNCs: Cellulose nanocrystals

In this study, various concentrations of CNCs were added to GIC samples. The right concentration can provide the ideal interaction between the crystal and cement matrix during a chemical reaction with the reinforcing structure formation.[12] As demonstrated previously, the concentration and intrinsic character of the reinforcing agent added to GIC affected the hardness of the resulting matrix.[4] In this study, the addition of CNCs as a reinforcing agent significantly increased the Vickers hardness value of the tested samples.

It is important to analyze the hardness of dental materials to ensure their clinical function, including resistance to masticatory forces.[18] Hardness testing has frequently been used to evaluate the surface resistance of materials to plastic deformation caused by penetration.[18] Hardness was shown to be closely related to compressive, flexural, and wear properties.[19] In the present study, hardness testing revealed the highest hardness value (38.89%) in the GIC-CNCs 0.4% group, at concentrations of CNCs above 0.4%, more glass particles were found on the surface so the hardness value decreases. The same thing was also found in a previous study, which showed decreased in Vickers micro-hardness from GIC reinforced with CNC from eucalyptus wood due to the large number of glass particles on the surface of the GIC.[4] As demonstrated previously, the proportion of glass particles and polyacid affected the hardness of GIC, its proportion influenced the concentration of filler required to produce a material with a good hardness value.[19] A previous study showed that fewer glass particles on the surface of GIC resulted in a low glass to polyacid ratio, which led to an increased ability of polyacids reaction with the nanoparticles.[20] In the same study, interstitial packing of these nanoparticles resulted in a higher nanoparticle: Matrix ratio at the interface, where the size constraints of the larger glass particles contributed to a surface layer rich

**Table 2: Statistical analysis of the differences in the Vickers hardness means among the six groups using a one-way analysis of variance and the Games-Howell *post hoc* test**

| Groups | GIC | GIC-CNCs 0.2% | GIC-CNCs 0.4% | GIC-CNCs 0.6% | GIC-CNCs 0.8% | GIC-CNCs 1% |
|---|---|---|---|---|---|---|
| GIC | - | 0.000* | 0.000* | 0.000* | 0.000* | 0.000* |
| GIC-CNCs 0.2% | - | - | 0.000* | 0.960 | 0.996 | 0.928 |
| GIC-CNCs 0.4% | - | - | - | 0.000* | 0.000* | 0.002* |
| GIC-CNCs 0.6% | - | - | - | - | 0.999 | 0.466 |
| GIC-CNCS 0.8% | - | - | - | - | - | 0.689 |
| GIC-CNCs 1% | - | - | - | - | - | - |

**P*<0.05: Statistically significant difference. GIC: Glass ionomer cement, CNCs: Cellulose nanocrystals in the matrix. Furthermore, larger glass particles size led to the more surface layer fill with matrix.[19]

The same study showed the integrity of the interface between glass particles and the matrix. In the present study, the increase in the micro-hardness value of the GIC-CNCs 0.4% group indicates the interaction of the filler and the matrix, which results in an ideal proportion of glass particles and acids on the GIC surface to reacts with the nanoparticles.

## Conclusion

The addition of CNCs from sugarcane bagasse significantly increased the Vickers hardness of GIC restorative material. Further studies are needed to investigate the bonding ability of GIC-containing CNCs from sugarcane bagasse with the tooth structure, its fluoride release ability, and biocompatibility, including clinical trials to further understand the properties and characteristics of this material, especially in the oral environment.

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### Conflicts of interest

There are no conflicts of interest.

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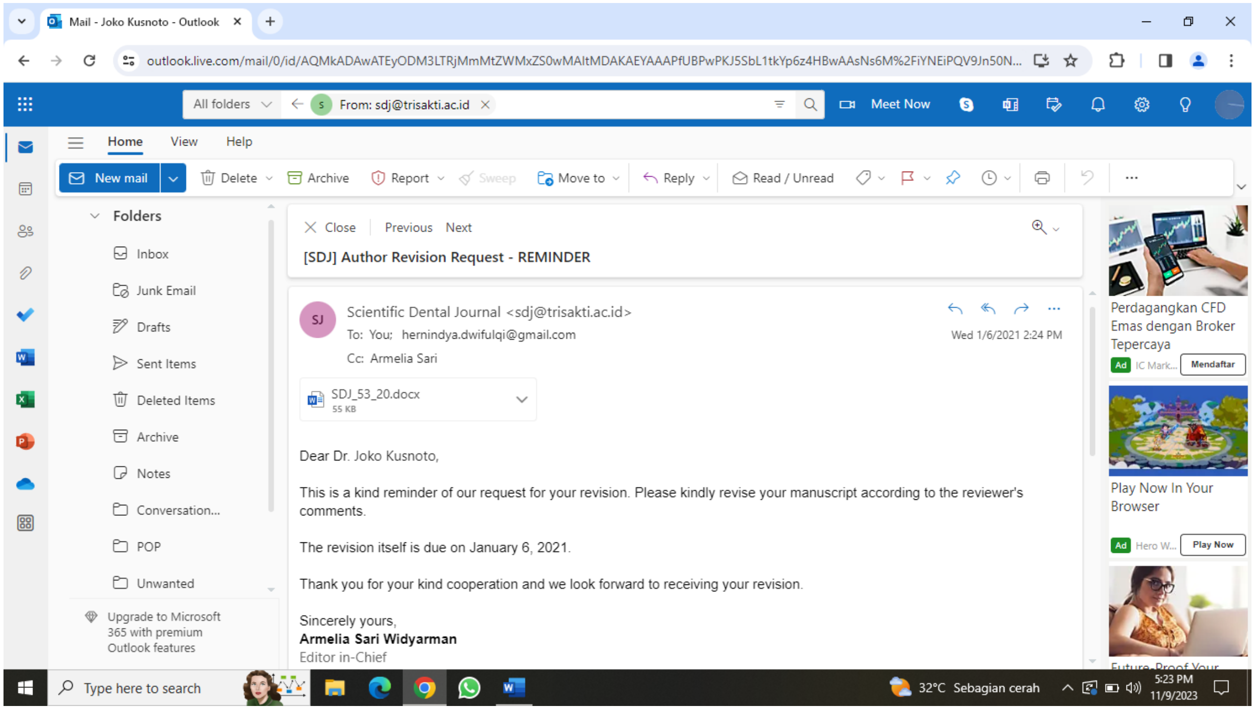

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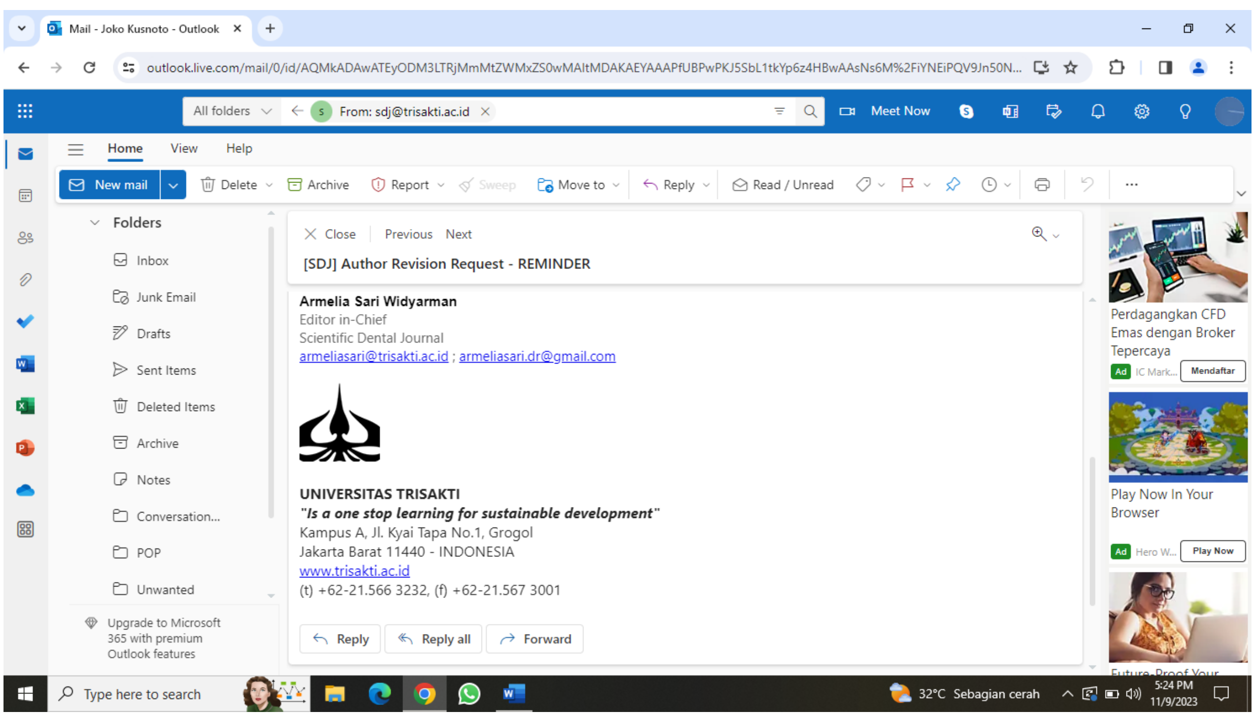

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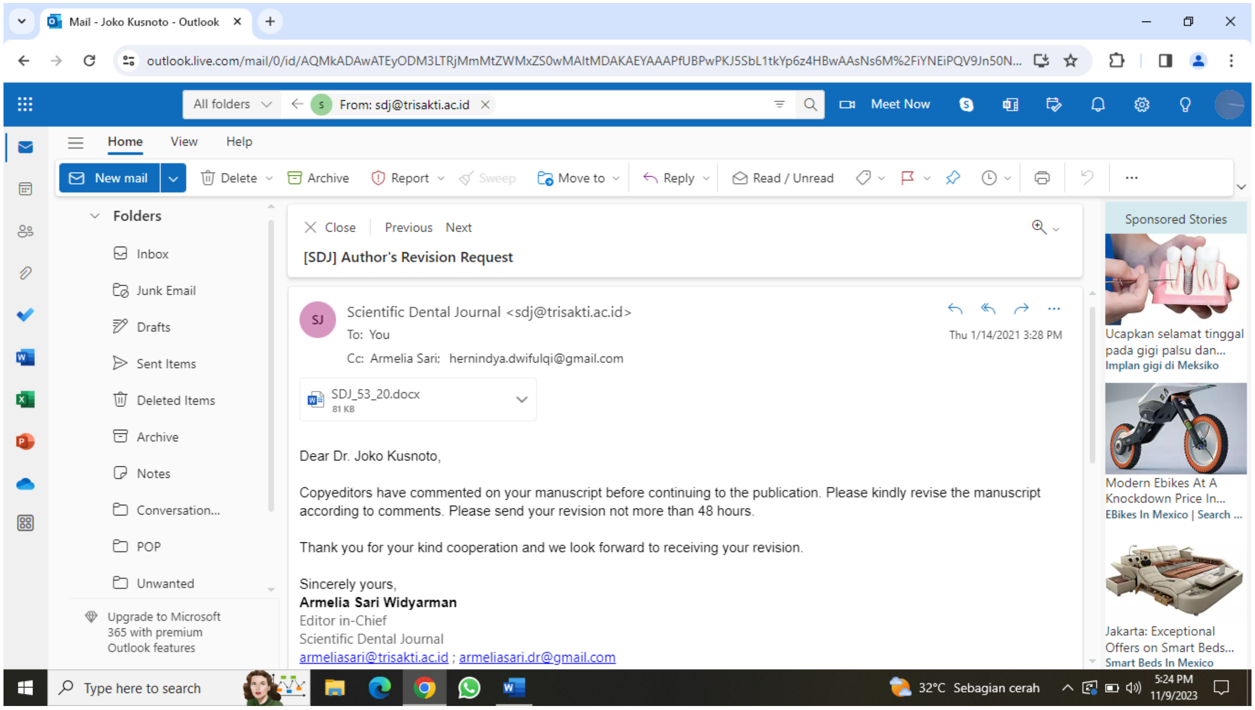
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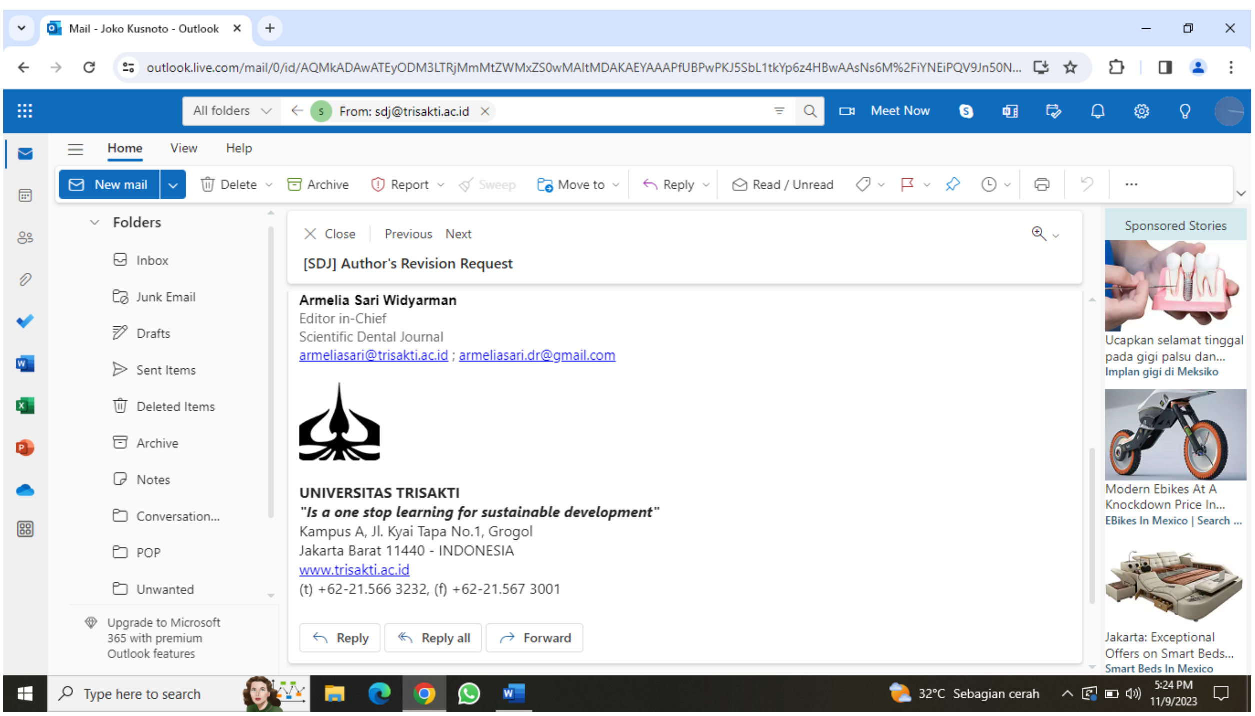
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