



Effect of Durian Rind Nanocellulose Addition on the Vickers Hardness of Self-Cure Acrylic Resin Fabricated By Spray-on Method

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ABSTRACT

*Self-cure acrylic resin is commonly used to fabricate orthodontic baseplates in removable appliances. However, it exhibits relatively low hardness, making it susceptible to wear and deformation under masticatory forces. The addition of reinforcing materials can enhance mechanical properties such as hardness. Nanocellulose derived from durian (*Durio zibethinus* Murr.) rind, an agricultural waste product, is a promising bio-based reinforcing material. This study aimed to determine the effect of durian rind nanocellulose addition on the Vickers hardness of self-cure acrylic resin fabricated using the spray-on method. This laboratory experimental study employed a posttest-only control group design. Durian rind nanocellulose was synthesized via acid hydrolysis and characterized using Transmission Electron Microscopy (TEM) and*

Fourier Transform Infrared Spectroscopy (FTIR) to confirm morphology and functional groups before incorporation into the acrylic resin. The nanocellulose was dispersed in monomer using ultrasonic homogenization for 5 minutes to ensure uniform distribution, then applied to polymer powder using the spray-on method. Twenty-four samples were divided into three groups (n=8): 2% nanocellulose, 3% nanocellulose, and control (0% nanocellulose). The highest mean hardness value was found in the 2% nanocellulose group (30.77 ± 1.03 VHN), while the lowest was in the control group (26.90 ± 0.79 VHN). Data were normally distributed and homogeneous (Shapiro-Wilk and Levene tests). One-way ANOVA revealed significant differences among groups ($p < 0.05$), with post hoc LSD tests confirming significant pairwise differences. This study concludes that durian rind nanocellulose addition significantly increases the Vickers hardness of self-cure acrylic resin fabricated by the spray-on method compared to the control group.

1. INTRODUCTION

The use of removable orthodontic appliances is an effective and efficient method to solve mild crowding malocclusion (Luther *et al.*, 2013). One essential component of removable orthodontic appliances is the baseplate, which is primarily fabricated from self-cure acrylic resin. Self-cure acrylic resins, mainly composed of poly(methyl methacrylate) (PMMA), are widely used in the fabrication of removable orthodontic appliances due to their ease of manipulation and favorable working properties (Esmaeilzadeh *et al.*, 2022).

Various methods can be employed to fabricate removable orthodontic baseplates. Two commonly used manipulation techniques are the spray-on (salt and pepper) method and the doughing method. The spray-on method, in which the polymer powder is saturated by its liquid monomer through a layering technique, has gained popularity in orthodontic clinics because it is easier to apply and requires less technical skill than the dough method (Ica *et al.*, 2014). However, despite its advantages in clinical application, the spray-on method may affect the final mechanical properties of the acrylic resin, including its hardness, due to variations in polymer-monomer mixing and polymerization conditions. One of the mechanical properties of self-curing acrylic is hardness. Hardness is a critical mechanical property of self-cure acrylic resin, as it describes

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the material's resistance to surface deformation under applied load. Surface hardness is closely related to abrasion, erosion, and attrition of restorative materials in the oral cavity. Inadequate hardness can result in the acrylic resin being unable to withstand the pressure produced during mastication, leading to cracking, wear, and eventual failure of the orthodontic appliance (Hameed and Rahman, 2015).

The increasing use of acrylic resin in the dental field has led to various efforts to enhance its mechanical properties. One approach is the addition of nanocellulose as a reinforcing agent (Evelyna et al., 2015). Nanocellulose has several advantageous properties, including low density, high surface area, ease of chemical or mechanical modification, excellent biocompatibility, and biodegradability (Thomas *et al.*, 2017). These properties make nanocellulose an attractive candidate for reinforcing dental materials.

Nanocellulose can be synthesized from durian (*Durio zibethinus* Murr.) rind, specifically from its epicarp layer (Zeni et al., 2015). Durian rind is an agricultural waste product that is abundantly available in Southeast Asian countries. The epicarp layer of durian rind contains high cellulose content (50-60%), along with hemicellulose (13%), lignin (15%), and starch (5%) (Kurniawan et al., 2013), making it a promising source for nanocellulose production. The utilization of durian rind for nanocellulose synthesis not only provides a sustainable and cost-effective reinforcing material but also contributes to waste management and environmental conservation.

Despite the potential benefits of nanocellulose as a reinforcing agent, information regarding the effect of durian rind nanocellulose addition on the hardness of self-cure acrylic resin orthodontic baseplates fabricated using the spray-on method remains limited. The spray-on method presents unique challenges in terms of nanocellulose distribution and polymerization behavior, which may influence the final mechanical properties differently compared to other fabrication methods. Understanding the relationship between the spray-on fabrication method and the reinforcing effect of nanocellulose is crucial for optimizing the mechanical performance of orthodontic baseplates. Therefore, this research aims to determine the effect of adding durian rind nanocellulose on the Vickers hardness of self-cure acrylic resin fabricated using the spray-on method. The findings of this study may contribute to the development of improved orthodontic materials with enhanced mechanical properties and better clinical performance.

2. METHOD

This study consists of three tests, Transmission Electron Microscopy (TEM), Fourier Transform Infrared Spectroscopy (FTIR) and Vickers Hardness test. Ethical eligibility permit from the Ethics Commission for Medical and Health Research, Faculty of Medicine, Jenderal Soedirman University with registration number 194/KEPK/IX/2020. Durian rind nanocellulose was synthesized by acid hydrolysis method and characterized by TEM and FTIR test. TEM and FTIR tests were conducted to confirm the morphology and functional groups of synthesized nanocellulose before being incorporated into the acrylic resin. TEM analysis was performed to determine the size and shape of the nanocellulose particles, while FTIR analysis was used to identify the chemical composition and functional groups present in the nanocellulose structure.

Twenty-four self-cure acrylic resin samples were prepared and divided into three groups using simple random sampling: (1) acrylic resin with 2% durian rind nanocellulose addition, (2) acrylic resin with 3% durian rind nanocellulose addition, and (3) acrylic resin without nanocellulose addition (control group). Each group consisted of eight samples (n=8).

The specimens were prepared with dimensions of 20 mm × 15 mm × 8 mm according to American Dental Association (ADA) Specification No. 12 for denture base polymers. The nanocellulose was first dispersed in the monomer (methyl methacrylate) using ultrasonic homogenization for 5 minutes to ensure even distribution throughout the monomer phase. This homogenization step was critical to prevent agglomeration and ensure uniform dispersion of nanocellulose particles.

The spray-on technique was employed for sample fabrication. The packing process was initiated by adding the nanocellulose-dispersed monomer to the polymer powder using a layering

technique consisting of four successive layers. This spray-on method involved alternately sprinkling polymer powder and saturating it with the monomer-nanocellulose mixture, allowing each layer to partially polymerize before adding the next layer. The specimens were allowed to polymerize at room temperature ($23 \pm 2^\circ\text{C}$) under atmospheric pressure for 24 hours to ensure complete polymerization and stabilization of the material properties. Following polymerization, sample finishing was performed using a tungsten carbide bur at a speed of 15,000 rpm for 60 seconds per sample to achieve smooth, standardized surfaces suitable for hardness testing

The Vickers Hardness test was performed on all samples. A diamond pyramid indenter was applied to the midpoint of each specimen surface with a standardized load. The diagonals of the resulting square-shaped indentation were measured using a microscope, and the Vickers Hardness Number (VHN) was calculated for each specimen. Three measurements were taken per sample, and the average value was recorded. The normality of data distribution was assessed using the Shapiro-Wilk test, while the homogeneity of variance was evaluated using Levene's test. Since the data met the assumptions of normality and homogeneity, one-way Analysis of Variance (ANOVA) was performed to determine whether there were significant differences in hardness values among the three groups. When the ANOVA showed significant differences ($p < 0.05$), post hoc analysis using the Least Significant Difference (LSD) test was conducted to identify which specific groups differed significantly from each other. Statistical significance was set at $\alpha = 0.05$.

3. RESULT AND DISCUSSION

Result

The Transmission Electron Microscope test could determine the shape and size of durian rind nanocellulose. Figure 1 below is a picture of the results of TEM characterization with strong magnification. The results of TEM characterization showed that durian rind nanocellulose had a whisker shape and tended to experience agglomeration. The Transmission Electron Microscope characterization with magnifications to 150.000x and particle sizes ranging from a diameter of 7 - 20 nm and a length of 275 - 475 nm.

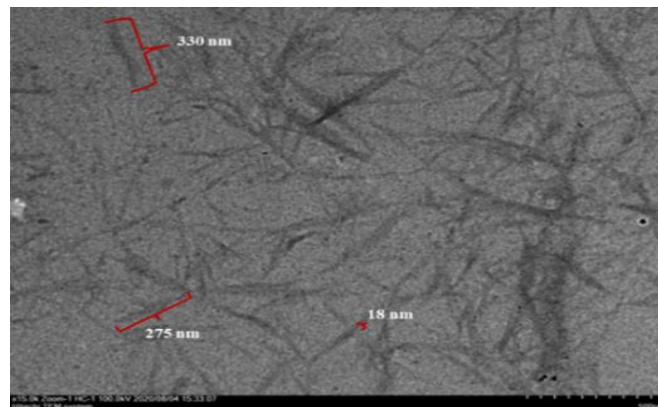


Figure 1. Transmission electron microscopic micrographs showing the shape and size of durian rind nanocellulose (magnification: 150,000 \times)

The chemical compositions of durian rind nanocellulose were examined by FTIR. Figure 2 below shows the FTIR spectra for durian rind nanocellulose. In the spectrum of the nanocellulose, the band in the spectrum which approximately near $1112,93 \text{ cm}^{-1}$ showed the present of C - O stretching vibration of acetyl group of lignin. The band around $1112,93 \text{ cm}^{-1}$ showed the present of C - O - C groups of ether. These particular peaks indicated the presence of starch in nanocellulose. The band in the spectrum which approximately near 896 cm^{-1} showed the present C - H. The band around $3408,22 \text{ cm}^{-1}$ showed the present of -OH groups. Moreover, the FTIR spectrum shows a peak of $1058,92 \text{ cm}^{-1}$ Si - O.

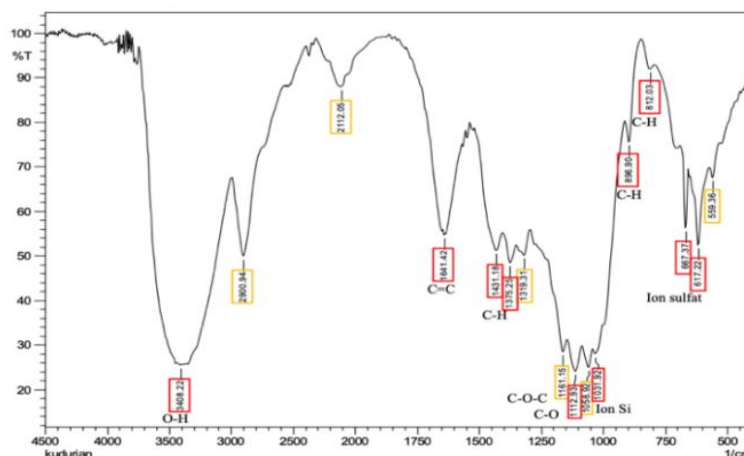


Figure 2. FTIR spectra of durian rind nanocellulose showing characteristic groups

Table 1 presents the mean values and standard deviations of Vickers Hardness Number (VHN) for self-cure acrylic resin samples in the three experimental groups. The highest mean hardness value was observed in the group with 2% nanocellulose addition (30.77 ± 1.03 VHN), followed by the 3% nanocellulose group (30.53 ± 0.81 VHN), while the lowest hardness was found in the control group without nanocellulose addition (26.90 ± 0.79 VHN).

Table 1. Mean and standart deviation of Vickers hardness number for self-curing resin acrylic

No	Sample groups	Mean (VHN) ± SD
1	2% Nanocellulose	30,77 ± 1,03
2	3% Nanocellulose	30,53 ± 0,81
3	Without Nanocellulose	26,9 ± 0,79

Prior to conducting the one-way ANOVA analysis, the normality and homogeneity of the data were assessed. The Shapiro-Wilk test confirmed that the data from all three groups were normally distributed ($p > 0.05$). Levene's test demonstrated that the variances among the groups were homogeneous ($p > 0.05$). These results indicated that the data met the assumptions required for parametric statistical analysis using one-way ANOVA.

The one-way ANOVA test results, presented in Table 2, revealed a statistically significant difference in hardness values among the three sample groups ($p = 0.000$, $p < 0.05$). This finding indicates that the addition of durian rind nanocellulose significantly affects the Vickers hardness of self-cure acrylic resin fabricated using the spray-on method.

Tabel 2. The One-Way ANOVA analysis

No	Sample groups	Sig.
1	2% Nanocellulose	0,000
2	3% Nanocellulose	
3	Without Nanocellulose	

To determine which specific groups differed significantly from each other, a post hoc Least Significant Difference (LSD) test was conducted. The results of the post hoc analysis are presented in Table 3. The LSD test revealed that all pairwise comparisons were statistically significant ($p < 0.05$). The comparison between the 2% nanocellulose group and the 3% nanocellulose group showed a marginally significant difference ($p = 0.590$), although both groups exhibited significantly higher hardness values compared to the control group. Both the 2% and 3% nanocellulose groups demonstrated significantly higher hardness values than the control group without nanocellulose addition ($p = 0.000$).

Table 3. Result of Post Hoc LSD hardness test of self-curing acrylic resin

Sample groups	2%	3%	without
2%		0,590	0,000*
3%			0,000*
Without			

Discussion

The TEM characterization confirmed that the synthesized durian rind nanocellulose exhibited whisker-like morphology with particle sizes in the nanometer range (diameter: 7-20 nm; length: 275-475 nm). These dimensions are consistent with nanocellulose specifications reported in previous studies, which typically range from 1 to 200 nm (Lee *et al.*, 2014). The whisker-like shape and nanoscale dimensions are crucial for reinforcement effectiveness, as they provide a high surface area-to-volume ratio that facilitates strong interfacial bonding with the acrylic resin matrix (Huang *et al.*, 2020; Barhoum *et al.*, 2021)

The FTIR spectra of the durian rind nanocellulose showed the present C – O of acetyl group of lignin, C – O – C groups of ether, C – H groups of alkaline, –OH groups and Si – O groups. This indicated that the acid hydrolysis treatment is suitable to produce cellulose at nano size scale. Apart from that, the nanocellulose have a great potential to be used as the reinforcement material for self-curing acrylic resin. This is because nanocellulose provides higher surface area (Rahman *et al.*, 2016).

The Vickers hardness test results revealed that both 2% and 3% nanocellulose additions significantly increased the hardness of self-cure acrylic resin compared to the control group. The group with 2% nanocellulose addition exhibited the highest mean hardness value (30.77 ± 1.03 VHN), representing approximately a 14% increase compared to the control group (26.90 ± 0.79 VHN). This finding is consistent with previous research demonstrating that nanocellulose incorporation can enhance the mechanical properties of polymer composites (Evelyna *et al.*, 2015). The mechanism by which nanocellulose enhances hardness can be attributed to several factors. First, nanocellulose particles can fill the interstitial spaces between the polymer chains of the acrylic resin, thereby increasing the material's density and resistance to deformation (Afrizal, 2016). The nanoscale dimensions of the cellulose particles allow them to penetrate and occupy these microscopic voids more effectively than larger reinforcing materials. Second, the high surface area of nanocellulose facilitates extensive interfacial bonding with the polymer matrix through hydrogen bonding and physical entanglement. These interfacial interactions create mechanical interlocking that inhibits the movement of polymer chains under applied load, thereby increasing the material's resistance to indentation (Shen *et al.*, 2020).

The finding that 2% nanocellulose concentration yielded slightly higher hardness than 3% concentration, although not statistically significant ($p=0.590$), suggests that there may be an optimal concentration for reinforcement effectiveness. This phenomenon can be explained by considering the balance between reinforcement and potential agglomeration. At higher concentrations, nanocellulose particles may have a greater tendency to agglomerate, reducing their effective surface area available for interaction with the polymer matrix. The TEM results in this study showed that the nanocellulose exhibited a tendency toward agglomeration, which supports this interpretation. When nanoparticles agglomerate, they lose some of their nanoscale advantages and may even act as stress concentration points, potentially compromising the mechanical properties. Therefore, the 2% concentration may represent a better balance between providing sufficient reinforcement and minimizing agglomeration effects. The improved hardness of self-cure acrylic resin with durian rind nanocellulose addition has important clinical implications for orthodontic appliances. Enhanced hardness suggests better resistance to wear, scratching, and surface deformation, which are common problems encountered during the clinical use of removable orthodontic appliances. Orthodontic baseplates are subjected to repeated insertion and removal, masticatory forces, and exposure to the oral environment. Improved hardness can extend the service life of these appliances, reduce the need for repairs or

replacements, and maintain better adaptation to the oral tissues over time. Additionally, harder surfaces are generally more resistant to biofilm accumulation and are easier to clean, which may contribute to better oral hygiene during orthodontic treatment.

The hardness of self-curing acrylic resin was influenced by several factors, one of which was the particle size. Nanocellulose could fill the space between the atoms of self-curing acrylic resin and inhibits the rate of dislocation of heat-cured acrylic resin atoms, thereby increasing the density and mechanical properties of self-curing acrylic resin (Afrizal, 2016). The nanocellulose that fills the space between the atoms of the self-curing acrylic resin is a mechanical bond. The use of nanofillers with a particle size of 10 – 100 nm can increase mechanical properties such as hardness, so that the use of removable orthodontic appliances with a sufficient period long expected to withstand abrasion (Rodrigues et al., 2008).

From a sustainability perspective, the use of durian rind nanocellulose as a reinforcing agent offers several advantages. Durian is widely cultivated in Southeast Asian countries, and its rind constitutes a significant portion of agricultural waste. By utilizing this waste material to produce nanocellulose for dental applications, this approach contributes to circular economy principles and reduces environmental burden. Furthermore, nanocellulose is a renewable, biodegradable, and biocompatible material, making it an attractive alternative to synthetic reinforcing agents that may have environmental and health concerns.

Despite the promising results, this study has several limitations that should be acknowledged. First, this study focused solely on hardness as a mechanical property. Other important properties such as flexural strength, impact strength, and surface roughness were not evaluated. Future studies should investigate the comprehensive mechanical properties of nanocellulose-reinforced self-cure acrylic resin to provide a more complete understanding of its clinical performance. Second, this study was conducted under laboratory conditions and did not simulate the complex oral environment, including exposure to saliva, temperature variations, and chemical agents. In vivo or simulated oral environment testing would be beneficial to assess the long-term performance and stability of the nanocellulose-reinforced material. Future research could explore alternative dispersion techniques or surface modification of nanocellulose to improve its compatibility with the acrylic resin matrix and reduce agglomeration.

4. CONCLUSION

This study concludes that the addition of durian rind nanocellulose significantly increases the Vickers hardness of self-cure acrylic resin fabricated using the spray-on method. The optimal enhancement was observed at a 2% nanocellulose concentration, yielding a hardness value of 30.77 ± 1.03 VHN compared to 26.90 ± 0.79 VHN in the control group without nanocellulose. The TEM and FTIR characterizations confirmed that the synthesized nanocellulose possessed appropriate morphological and chemical properties for reinforcing acrylic resin.

5. ACKNOWLEDGE

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