

Tensile and Flexural Properties of Epoxy Nanocomposites Reinforced with Cellulose Nanocrystals

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Abstract

Composite materials are extensively utilized across various fields such as engineering, aviation, automotive, construction, and healthcare. This widespread application highlights their superior properties, often absent in the individual constituent materials. Additionally, composites offer the advantage of being easily fabricated to meet specific requirements, and incorporating natural fibers as reinforcement has gained significant interest due to their environmental friendliness and abundance. Nanocellulose is a promising green material due to its unique characteristics. Specifically, cellulose nanocrystals (CNC), nanoscale derivatives of nanocellulose, have attracted considerable attention as reinforcement agents in composite fabrication. This interest stems from CNC's notable advantages, including excellent mechanical properties, a high crystallinity index, plentiful availability, low weight, and eco-friendly nature. This study was undertaken to investigate the impact of varying concentrations of cellulose nanocrystal (CNC) (0, 0.5, 0.75, 1 wt%) on the mechanical properties, specifically the tensile and flexural properties, of epoxy resin/cellulose nanocrystal (E/CNC) nanocomposites. The materials employed in this research include epoxy resin, hardener, and cellulose nanocrystals. The fabrication of the E/CNC nanocomposites was carried out through a straightforward mixing method, wherein the constituent materials were blended following the defined experimental parameters, followed by the molding process. The findings of this study indicate that the incorporation of cellulose nanocrystals (CNC) significantly enhances the mechanical properties of E/CNC nanocomposites. The E/0.75CNC nanocomposite showed optimal tensile strength (39.91 MPa; +4.95%), while E/1CNC exhibited superior flexural strength (65.78 MPa; +5.08%) compared to the unmodified epoxy baseline.

Keywords: Cellulose Nanocrystal, Epoxy, Nanocomposite, Mechanical Properties, Tensile Strength.

1. Introduction

Composite materials have been extensively employed in everyday life across diverse fields such as engineering, aviation, automotive, construction, and healthcare [1] [2] [3] [4]. This widespread use demonstrates the significant benefits of composite materials, particularly their enhanced properties that are not inherent in the individual constituent components. Various methods exist for manufacturing composites, among which molding is a common technique. This process involves pouring the matrix material into a mold shaped according to the desired form and specifications. The primary advantages of molding include its simplicity and suitability for large-scale production [3] [5] [6] [7] [8] [9] [10]. Generally, composite materials have two main components: a matrix and reinforcement [11][12]. Epoxy (E) is one type of thermoset polymer widely used as a matrix in composites [13]. This is because epoxy has relatively greater strength and stiffness than other polymers [9], [11]. In addition, epoxy resin also has good temperature resistance, low shrinkage power, resistance to air humidity, chemical resistance, and pressure resistance [1][14][15][16][17]. However, epoxy resins are brittle and susceptible to crack propagation [17]; one approach to enhancing the mechanical properties of epoxy matrices involves incorporating reinforcing materials at the nanometer scale, which can significantly contribute to the composite's overall performance and structural integrity.

With increasing attention to environmental and sustainability issues, the use of environmentally friendly and biodegradable reinforcing materials is increasingly in demand [18][19][20]. Nanocellulose is one of the promising green materials because of its unique properties [21][22]. Cellulose nanocrystals (CNC), a form of nanocellulose, have garnered significant interest due to their potential as a highly effective reinforcement material in producing advanced composites, owing to their remarkable mechanical properties and sustainability advantages. [2][16][23][24]. This is because CNC has several advantages, including good mechanical properties, high crystallinity index,



good binding ability, strong against heat, insoluble in water, lightweight, and environmentally friendly [6][7][8][25][26][27]. Cellulose nanocrystals (CNCs) are a class of advanced nanomaterials derived from cellulose, a natural polymer abundantly present in the cell walls of plants. Among the various methods employed to isolate CNCs from cellulose, the acid hydrolysis process stands out as one of the most widely utilized and effective techniques, as it selectively removes the amorphous regions of cellulose, thereby yielding the highly crystalline nanostructures [5][28][29]. CNCs have a size of nanometers (10^{-9} meters) and a rod- or needle-like shape [21][22].

Therefore, nanocellulose is an attractive reinforcement material for matrices in composite materials. According to the literature review, research on the impact of nanocellulose addition to epoxy matrices remains limited. Given the promising characteristics of cellulose nanocrystals (CNC), it is essential to investigate how varying CNC content influences the mechanical properties of epoxy/CNC composites. In this study, the mechanical behavior of these composites was evaluated through tensile and flexural testing, with CNC content varied at 0, 0.5, 0.75, and 1 wt% [17].

2. Research Methods

2.1. Materials

The cellulose nanocrystals (CNC) employed in this investigation were commercially procured from Nanografi, based in Turkey. These CNCs possess a density of 1.49 g/cm^3 , with their length varying between 300 and 900 nm and their width ranging from 10 to 20 nm. Additionally, the CNCs exhibit a high % crystallinity index of 92% and demonstrate a thermal decomposition temperature of 349°C . The epoxy resin utilized in this study is the Eposchon A brand, a Bisphenol A–Epichlorohydrin type, which was combined with the Eposchon B hardener, a Polyaminoamide type, in a 2:1 resin-to-hardener ratio. Both materials were sourced from PT. Justus Kimia Raya in Semarang.

2.2. Preparation of epoxy/cellulose nanocrystal (CNC) nanocomposites

The epoxy/CNC nanocomposite manufacturing process was made using simple mixing and continued with molding, where the research materials will be mixed according to the research parameters, as shown in Figure 1. Cellulose nanocrystal (CNC) was preheated using an oven at 60°C for 2 hours to reduce the moisture content still contained in the CNC. The epoxy matrix was carefully blended with cellulose nanocrystals (CNC) at concentrations of 0.5, 0.75, and 1 wt% using a mechanical stirrer set to 500 rpm while simultaneously being heated to 70°C with a magnetic stirrer for 4 hours. Following this, the epoxy/CNC mixture was combined with the hardener and stirred for 3 minutes to ensure a homogeneous consistency. Subsequently, a vacuum was applied for 10 minutes to the epoxy/CNC/hardener blend to eliminate any entrained gas molecules. The resultant mixture was then evenly poured into an acrylic mold and left to cure for 24 hours at ambient room temperature. A mirror glaze was applied to facilitate the smooth removal of the nanocomposite samples from the mold. A similar procedure was employed to prepare pure epoxy composites without adding CNC reinforcement.

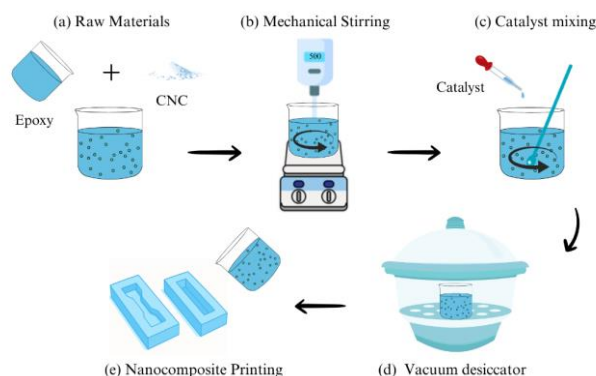


Fig 1. Epoxy/CNC Nanocomposite Manufacturing Process

2.3. Mechanical testing

The tensile testing of the nanocomposites was carried out following the ASTM D638-02 (Type IV) standard, employing a crosshead speed of 2 mm/min. In contrast, the flexural testing complied with the ASTM D7264 standard, using a crosshead speed of 2 mm/min. These tests were conducted utilizing a universal testing machine (UTM) of the Zwick/Roell Z020 model to evaluate the mechanical properties, specifically the tensile and flexural properties, of the epoxy/CNC nanocomposites. Five samples were subjected to testing for each testing condition, and the resulting data were subsequently averaged to ensure reliable and representative results.

3. Result and Discussion

3.1. Tensile Properties

Figure 2 provides a visual representation of the influence of CNC incorporation on the tensile strength of epoxy/CNC nanocomposites. The tensile strength of the pure epoxy was determined to be 38.02 MPa, whereas the introduction of CNC reinforcement led to a notable enhancement in the tensile strength. These results underscore the reinforcing effect of CNC on the epoxy matrix, indicating its ability to improve the material's mechanical properties. The maximum tensile strength was observed in the nanocomposites incorporating 0.75 wt% CNC, where a value of 39.91 MPa was recorded, showcasing the optimal reinforcement achieved at this specific concentration. The tensile strength of the E/0.75CNC nanocomposite exhibited a notable increase of 4.95%, a result attributed to the homogeneous dispersion of the nanometer-sized CNC within the epoxy matrix. This uniform distribution of CNC particles facilitates an even

distribution of the applied load across the nanocomposite, enhancing its overall mechanical performance. Similar results were also found in the research of Huang et al. [1], Li et al. [15], and Zhu et al. [4], where the addition of CNC with different variations in the matrix can increase the tensile strength and tensile modulus. This enhancement can be attributed to the uniform dispersion of CNC within the matrix and the effective interaction between the CNC particles and the epoxy matrix, which collectively ensures the even distribution of stress throughout the nanocomposite structure. Research by DiLoreto et al. [26] showed different results, where the addition of CNC did not cause tensile strength in the matrix. The tensile strength of the E/0.5CNC nanocomposite increased by 2.23%, while that of the E/1CNC nanocomposite increased by 1.89%. When compared to E/0.75CNC nanocomposites, the decrease in tensile strength that occurs in E/0.5CNC nanocomposites and E/1CNC nanocomposites is due to the presence of CNC agglomeration in the epoxy matrix, causing stress concentration in one part. In addition, the cause of the decrease and not maximizing the tensile strength of epoxy/CNC nanocomposites can also be influenced by the aspect ratio of CNC, the hydrophilic nature of CNC, and porosity. Several similar studies on cellulose nanocrystal (CNC) have also been conducted using other types of matrices such as PSF [28], PBDS [19], UPR [3], PVA [20], PES [29], PLA [8], and NR [2]. They reported that the addition of CNC can increase the composites' tensile strength and tensile modulus.

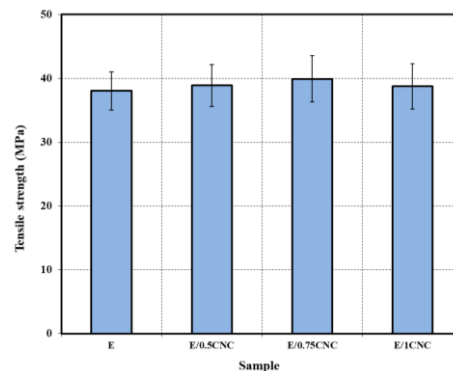


Fig 2. Effect of CNC weight fraction on tensile strength (E, E/0.5CNC, E/0.75CNC, and E/1CNC)

Figure 3 illustrates the impact of CNC incorporation on the tensile modulus of epoxy/CNC nanocomposites. The results mirror the trends observed in the tensile strength of the nanocomposites. Pure epoxy exhibits a tensile modulus of 1329 MPa; however, the introduction of CNC reinforcement into the epoxy matrix has led to a notable increase in the tensile modulus. The highest tensile modulus was observed in the E/0.75CNC nanocomposite, which demonstrated a 5.03% improvement, rising from 1329 MPa to 1396 MPa, thereby indicating the enhancement of the material's rigidity through CNC incorporation. This improvement can be attributed to the enhanced dispersion of CNCs within the epoxy matrix and the strengthened interfacial bonding between the CNCs and the epoxy.

Furthermore, the observed decrease in elongation at break in the E/CNC nanocomposites indicates the increasing stiffness of the material. As the material becomes stiffer, its tensile modulus is correspondingly elevated. Although a reduction in tensile modulus is observed in the E/0.5CNC and E/1CNC nanocomposites, their tensile modulus values remain higher than that of the pure epoxy, suggesting the continued reinforcement effect of CNC incorporation. This is attributed to the agglomeration of CNCs on the epoxy matrix. The properties of the epoxy matrix and CNC determine the increase and the usage ratio between the matrix and reinforcement. Research by Huang et al. [1] reported that adding 5 wt% CNC into the epoxy matrix found the highest tensile modulus. This indicates a good interaction between the epoxy matrix and CNC. Research by DiLoreto et al. [26] also reported an increase in tensile modulus and showed different results with decreased tensile strength.

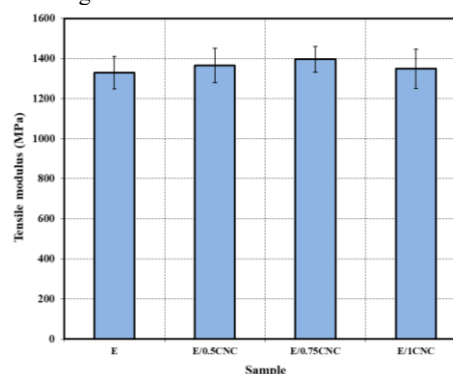


Fig 3. Effect of CNC weight fraction on tensile modulus (E, E/0.5CNC, E/0.75CNC, and E/1CNC)

Figure 4 presents the elongation results at break observed during the tensile testing of epoxy/CNC nanocomposites. The elongation at break is measured at 3.45% for the epoxy matrix without CNC reinforcement. Upon incorporating CNC into the epoxy matrix, a reduction in elongation at break is observed, suggesting that the addition of CNC does not enhance the ductility of the nanocomposite. Compared to the pure epoxy matrix, a decrease in elongation at break is particularly evident in the E/0.5CNC and E/1CNC nanocomposites. The lowest elongation at break is recorded in the E/1CNC composite, where a reduction of 8.75% is noted, with the value decreasing from 3.45% to 3.15%. This indicates a stiffening effect induced by CNC incorporation. The decrease in elongation at break was attributed to CNC agglomeration and poor interfacial bonding. Similar results were also found in the research of DiLoreto et al. [26], where the addition of CNC did not show an increase in elongation at break in the composite material.

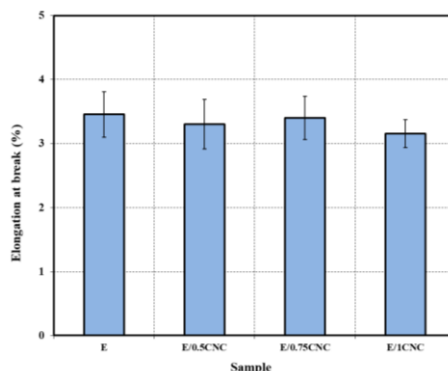


Fig 4. Effect of CNC weight fraction on elongation at break (E, E/0.5CNC, E/0.75CNC, and E/1CNC)

The tensile test results revealed increased strength due to the addition of cellulose nanocrystals (CNC) into the epoxy matrix. However, this improvement was not particularly significant compared to several previous studies. This outcome can be attributed to various factors discussed earlier. Several studies have also reported that cellulose nanocrystals (CNC) can be nanofillers in composite materials reinforced with natural or synthetic fibers. CNCs enhance interfacial bonding, thereby strengthening the interaction between the matrix and the reinforcement, resulting in improved mechanical properties. This advantage is closely related to the inherent characteristics of CNCs.

3.2. Flexural Properties

Figure 5 shows the effect of CNC addition on the flexural strength of epoxy/CNC nanocomposites. Epoxy has a flexural strength of 62.60 MPa, and adding CNC (0.5, 0.75, and 1 wt%) to the epoxy matrix can increase the flexural strength. The highest flexural strength is found in the E/1CNC nanocomposite, which is 65.78 MPa, with an increase of 5.08%. This increase occurs because CNC can be dispersed evenly. The addition of CNC affects the bonding interface because CNC has a high aspect ratio and crystallinity content, which will cause an increase in strength in the epoxy matrix. The research results by Maradini et al. [27] also show that adding 2 wt% CNC can increase flexural strength by 159%. This can be attributed to the efficient dispersion of CNC in the matrix without air voids. The increase in flexural strength in E/0.5CNC nanocomposites was 1.18%, and in E/0.75CNC nanocomposites was 2.96%. The bond between the surface of the reinforcing material and the matrix determines the strength of a composite. The bond formed depends on the nature of the matrix and reinforcing material used, the ratio of reinforcing material to matrix, geometry, and fiber orientation in the composite. Research by DiLoreto et al. [26] showed a decrease in flexural strength. The addition of CNC does not affect the matrix's flexural strength due to agglomeration.

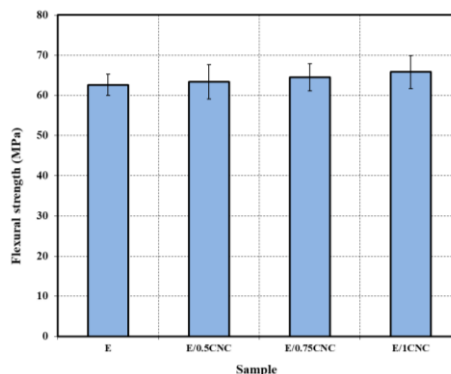


Fig 5. Effect of CNC weight fraction on flexural strength (E, E/0.5CNC, E/0.75CNC, and E/1CNC)

Figure 6 also shows the flexural modulus results of the E/CNC nanocomposites. The results obtained are similar to the flexural strength of the nanocomposites. Epoxy has a flexural modulus of 2041 Mpa, and the addition of CNC to the epoxy matrix shows an increase in modulus. The E/1CNC composite has the highest flexural modulus of 2164 MPa, with a rise of 6.03%. The flexural modulus of the E/0.5CNC nanocomposite has an increase of 2.69%, while the E/0.75CNC nanocomposite is 4.31%. The increase occurs because the CNC is well dispersed and evenly distributed in the matrix, thus forming a strong interaction between epoxy and CNC. These results show that composites containing 1 wt% CNC have the highest flexural modulus and flexural strength values. The decrease in flexural modulus can be caused by CNC agglomeration and high porosity [27], [28]. The flexural test results also indicate an improvement in the epoxy matrix due to the addition of cellulose nanocrystals (CNC). However, the observed enhancement was not particularly significant compared to previous studies' findings.

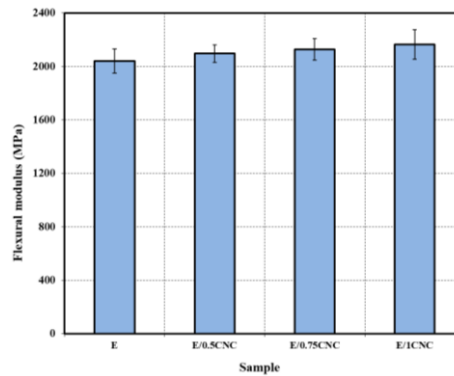


Fig 6. Effect of CNC weight fraction on flexural modulus (E, E/0.5CNC, E/0.75CNC, and E/1CNC)

4. Conclusion

The results indicate that the addition of cellulose nanocrystals (CNC) effectively enhances the nanocomposites' tensile and flexural properties. The optimal CNC content for improving tensile strength was observed in the E/0.75CNC nanocomposite, while the best flexural performance was achieved in the E/1CNC nanocomposite. The E/0.75CNC nanocomposite exhibited a tensile strength of 39.91 MPa, representing a 4.95% increase, and a tensile modulus of 1396 MPa, corresponding to a 5.03% improvement. Meanwhile, the E/1CNC nanocomposite demonstrated a flexural strength of 65.78 MPa with a 5.08% increase and a flexural modulus of 2164 MPa, reflecting a 6.03% enhancement. Due to their nanometer scale, CNCs can be uniformly and well-dispersed within the epoxy matrix, thereby increasing the interfacial surface area. This uniform dispersion suggests a strong interaction or bonding between the epoxy matrix and CNC. Furthermore, the strength of this bond depends on factors such as the nature of the matrix and reinforcement materials, the ratio of reinforcement to the matrix, and the geometry and orientation of the fibers within the composite. Additionally, porosity introduced during the fabrication process can influence the tensile and flexural properties of the nanocomposites.

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