



Influence of carbon nanotubes on physical and mechanical properties of polypropylene/reed flour hybrid nano WPC

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ABSTRACT: This study investigated the effect of incorporating multi-walled carbon nanotubes (MWCNTs) into polypropylene/reed flour (PP/RF) composites, aiming to enhance their mechanical and physical performance. Composites were prepared using various MWCNT concentrations (0, 1, 2, 3, and 5 phr) and characterized through mechanical testing, water absorption analysis, and scanning electron microscopy (SEM). The addition of MWCNTs significantly improved flexural and tensile properties, with peak tensile strength observed at 2 phr (approximately 15% higher than the control) and maximum flexural strength at 3 phr (approximately 15% higher). Impact resistance was also highest at 2 phr (around 10% increase compared to control). Although mechanical performance declined at 5 phr due to MWCNT agglomeration, this formulation exhibited the lowest water absorption (approximately 12% reduction) and thickness swelling, indicating improved hydrophobicity. SEM micrographs confirmed better filler dispersion at lower concentrations and agglomerate formation at higher loadings. The results highlight the reinforcing potential of MWCNTs, particularly at 2-3 phr, in improving both structural strength and moisture resistance.

Keywords: WPC, reed flour, carbon nanotube, mechanical properties, water absorption

Polipropilen/kamış unu hibrit nano WPC'nin fiziksel ve mekanik özellikleri üzerine karbon nanotüplerin etkisi

ÖZ: Bu çalışmada, çok katmanlı karbon nanotüplerin (MWCNT) polipropilen/kamış unu (PP/RF) kompozitlerine katılmasının mekanik ve fiziksel performans üzerine etkisi araştırılmıştır. Kompozitler, farklı MWCNT oranlarıyla (0, 1, 2, 3, ve 5 phr) hazırlanmış ve mekanik testler, su emme analizi ve taramalı elektron mikroskobu (SEM) ile karakterize edilmiştir. MWCNT ilavesi, özellikle eğilme ve çekme özelliklerinde belirgin iyileşmeler sağlamıştır; çekme mukavemeti 2 phr'de (yaklaşık 15% artış), eğilme mukavemeti ise 3 phr'de (yaklaşık 15% artış) en yüksek düzeye ulaşmıştır. Darbe dayanımı da 2 phr'da en yüksekti (kontrol ile karşılaştırıldığında yaklaşık %10 artış). MWCNT aglomerasyonu nedeniyle mekanik performans 5 phr'de düşmesine rağmen, bu formülasyon en düşük su emilimini (yaklaşık %12 azalma) ve kalınlık şişmesini göstererek hidrofobisitede iyileşme gösterdi. SEM mikrografları, daha düşük konsantrasyonlarda daha iyi dolgu dağılımı ve daha yüksek yüklemelerde aglomera oluşumunu doğruladı. Sonuçlar, MWCNT'lerin özellikle 2-3 phr'de hem yapısal dayanıklılığı hem de nem direncini iyileştirmede güçlendirme potansiyelini vurgulamaktadır.

Anahtar kelimeler: Polipropilen kompozit, kamış unu, karbon nanotüp, mekanik özellikler

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1 Introduction

Composite materials are engineered by combining two or more constituents in such a way that the resulting material exhibits enhanced properties compared to its individual components. Typically, one of the substances acts as the matrix, which binds the structure together while the others serve as reinforcements such as fibers or particles (Saarela et al., 2007). These materials often outperform traditional ones in terms of mechanical characteristics, offering a favorable strength-to-weight ratio and environmental sustainability (Vuorinen, 2007).

The mechanical behavior of fiber-reinforced composites is largely influenced by factors such as the size and orientation of fibers, their distribution, the nature of bonding at the fiber-matrix interface, and the volume and aspect ratio of fibers (Siró and Plackett, 2010).

In recent years, wood/natural fiber plastic composites (WPCs) have gained considerable attention. These composites are made by embedding organic or inorganic fillers into a thermoplastic matrix such as polyethylene (PE), polypropylene (PP), or polyvinyl chloride (PVC). Wood-based fillers offer notable benefits over conventional fillers like glass fibers or minerals due to their low cost, light weight, renewability, and minimal abrasiveness to machinery. WPCs are increasingly utilized in applications ranging from construction and automotive parts to infrastructure (Selke and Wichman, 2004; Klyosov, 2004).

Ongoing research focuses on optimizing the performance of WPCs through adjusting raw material compositions, improving wood-plastic interface adhesion, and refining processing parameters (Lu et al., 2000; Mohanty et al., 2001; Kim, 2012).

Hosseinihashemi et al., (2023, 2025) demonstrated that thermal and chemical-additive treatments of wood/plastic composites improve their durability and resistance, while Bal et al., (2023) reported that adding fillers such as wood flour or glass flour can optimize the mechanical behavior of polymer-based composites. These findings suggest that combining nano-additives with natural fillers can effectively improve the performance of hybrid wood-plastic composites.

Previous studies have investigated polypropylene (PP) composites reinforced with natural fillers such as reed flour and nano-additives like multi-walled carbon nanotubes (MWCNTs) to improve their viscoelastic properties. It has been shown that the addition of MWCNTs can enhance storage modulus, loss modulus, and creep resistance, while also reinforcing shear behavior. In fiber-reinforced PP/wood-flour composites, improvements in mechanical properties have been observed; however, challenges such as filler-matrix incompatibility, non-uniform dispersion of nanotubes, and increased viscosity remain. The current study combines MWCNTs with reed flour in PP matrices to address these issues and develop composites with improved viscoelastic performance (Kord et al., 2017).

Carbon nanotubes (CNTs), known for their extremely high Young's modulus, are recognized as highly effective reinforcements when dispersed in polymer matrices. Their inclusion in composites significantly boosts mechanical properties such as tensile strength and stiffness, making them suitable for high-performance applications (Coleman et al., 2006).

In addition to mechanical enhancements, CNTs contribute to improved energy absorption (Sun et al., 2009), abrasion resistance (Giraldo et al., 2008), as well as enhanced thermal and electrical conductivity (Huang et al., 2005; Hollertz et al., 2011). Their high aspect ratio, the ratio of length to diameter, is central to their reinforcing capability (Cataldo et al., 2009).

Currently, CNT-reinforced composites are used across industries, including automotive, aerospace, defence, electronics, energy, and sporting goods (Kingston et al., 2014). The synergy of lightweight, high surface area, and exceptional mechanical strength makes CNTs ideal for enhancing composite performance.

Specifically, multi-walled carbon nanotubes (MWCNTs) improve the interfacial bonding between wood fibers and polymer matrices, which is critical in WPCs. These nanostructures act as particulate fillers at the nanoscale, facilitating stress transfer and enhancing durability (Kim and Mai, 1998; Ma et al., 2010). As demonstrated by Jin and Matuana (2010), integrating CNTs into the shell layer of WPCs enhances flexural properties, although the inherent flexibility of PVC may influence the overall mechanical behavior. Recent studies (Park et al., 2020; Jian et al., 2022; Łukawski et al., 2023; Zhao et al., 2025) have shown that integrating carbon nanotubes and graphene into wood-plastic composites can significantly enhance mechanical strength, moisture resistance, and flexural behavior.

This study aimed to explore the use of reed as an alternative to traditional wood fillers in WPCs while also examining the effect of including MWCNTs on the mechanical and physical performance of polypropylene/reed flour nanocomposites. The specific objective was to identify the optimum MWCNT content (2-3 phr) that would maximise tensile, flexural, impact and water resistance properties.

2 Material and Method

2.1 Materials

Reed stalks were supplied by Anzali lagoon in the northern Iranian province of Gilan to prepare lignocellulosic materials. The reed stalks were cut to pieces 2 cm in length by a band saw. Then, they were milled with a hammer mill. The obtained reed flour was ground with a Thomas-Wiley miller to a fine powder of 40-60 mesh size, and then oven-dried and stored in sealed plastic bags before processing.

Polymer matrix comprises an injection molding grade polypropylene (PP) with the commercial name of Z30, they were supplied in the form of pellets by Marun Petrochemical Complex in Iran. The melt flow index (MFI) of the provided PP was 25 g/10 min and had a density of 0.9 g/cm³.

Coupling agent, maleic anhydride grafted polypropylene (PP-g-MA) in the form of granules was supplied by Solvey (Solvey International Chemical Group, Brussels, Belgium) with a trade name of Priex 20070 (MFI = 64 g/10 min, grafted Ma 1 wt%).

MWCNTs nanostructures introduced to the composites were supplied by Iran's Research Institute of Petroleum Industry (RIPI). The outer and inner diameters of the MWCNTs were about 30 and 10 nm, respectively, and the purity was 90%.

2.2 Sample preparation

Reed flour was initially dried in an oven at 85 °C for 24 h to prepare the composite specimens. Then, PP, reed flour (RF), coupling agent (CA), and MWCNTs were mixed and packed, in accordance to the mix proportions given in Table 1.

Composites were produced through a two-stage process. In the first step, PP was fed to the mixing chamber. After PP melted, the compatibilizer and MWCNTs were added. Reed flour was then fed through after passing for 5 minutes. The total mixing time was 13 min. The mixing was carried out with a co-rotating twin-screw extruder (Dr. Collin GmbH, Germany).

Table 1. PP/RF nanocomposites mix proportions

Samples label	Reed flour (RF) (wt.%)	Polypropylene (PP) (wt.%)	MWCNTs (phr)	Coupling agent (CA) (phr)
50-50-0	50	50	0	2
50-50-1	50	50	1	2
50-50-2	50	50	2	2
50-50-3	50	50	3	2
50-50-5	50	50	5	2

The barrel had five consecutive heated zones with temperatures set at 165, 170, 175, 180, and 185 °C, respectively. The screw speed was 60 rpm. The product was recovered by guiding the molten extrudate into a standard cold water stranding bath. The cooled strands were pelletized using a pilot scale grinder, dried and stored in sealed plastic bags. In the second stage, test specimens were injection molded by an injection molder at a molding temperature of 180 °C and an injection pressure of 4 MPa (Eman machine, Aslanian Co., Iran) to produce standard ASTM specimens. Finally, specimens were conditioned at a temperature of 20 °C and a relative humidity of 50% before testing according to ASTM D618.

2.3 Methods

2.3.1 Mechanical properties

Flexural and tensile strength assessments were conducted in accordance with ASTM D790 and ASTM D638 standards, respectively. An Instron testing machine (Model 8112, USA) was employed for these tests, with crosshead speeds set at 1 mm/min for flexural and 2 mm/min for tensile measurements. The Izod impact strength was evaluated at ambient temperature using a Santam pendulum impact tester (Model SIT-20D). Test specimens measured 62.5 mm × 13 mm × 3 mm and were notched to a depth of 2.5 ± 0.02 mm at a 45° angle, consistent with ASTM D256 guidelines. The results presented reflect the average values derived from four specimens per group.

2.3.2 Physical properties

Water absorption and thickness swelling of the composites were determined using ASTM D7031 procedures. Three specimens per composition were dried in an oven at 103 ± 2 °C for 24 hours. Their initial weights and thicknesses were measured with an accuracy of 0.001 g and 0.001 mm, respectively. The samples were immersed in distilled water at room temperature for 24 hours, then surface moisture was gently removed using paper towels, and post-immersion measurements were taken.

2.3.3 Statistical analysis

A one-factor analysis of variance (ANOVA) was used to evaluate the effects of different MWCNT concentrations. Duncan's multiple range test (DMRT) was employed at a 95% confidence level to distinguish significant differences among treatment means when ANOVA revealed statistical significance.

2.3.4 Scanning electron microscopy (SEM) characterization

Composite morphology was examined using field emission scanning electron microscopy (FE-SEM, TESCAN MIRA3 XMU, Czech Republic). A gold layer of approximately 25 nm was sputter-coated on the samples to mitigate charging during imaging. The specimens were analyzed under an accelerating voltage of 5 kV to investigate fracture surfaces and filler dispersion characteristics.

3 Results and discussion

The statistical analysis using ANOVA demonstrated significant effects of MWCNT incorporation on key mechanical and physical properties of the composites. The F-values for water absorption, thickness swelling, impact strength, flexural and tensile strength, and moduli are summarized in Table 2, indicating the degree of influence exerted by the nanofillers.

Table 2. F-value (in analysis of variance) obtained from statistical analysis

Investigated property	F-value
Water absorption (24 h)	7.147
Thickness swell (24 h)	3.578
Impact strength	3.143
Flexural strength	31.264
Flexural modulus	22.113
Tensile strength	803.185
Tensile modulus	728.611

3.1 Flexural and tensile strength

The mechanical enhancement of composites is influenced by multiple factors including fiber type, orientation, interfacial adhesion, and modulus of each component (Hull and Clyne, 1996). CNTs possess a Young's modulus around 1 TPa (Salvetat et al., 1999), and their integration into wood composites significantly boosts stiffness and tensile strength (Ma et al., 2010). Replacing wood fibers (5000-8000 MPa modulus) with CNTs can substantially improve elastic performance (Farsi and Sani, 2014). According to Hazarika and Maji (2014), CNTs interact chemically with hydroxyl and methylol groups in lignocellulosic components, resulting in improved composite strength and durability.

Figures 1 and 2 show the flexural and tensile properties as a function of MWCNT content. Maximum flexural strength and modulus were observed in specimens with 3 phr MWCNTs. In contrast, optimal tensile strength and modulus occurred at 2 phr. Both flexural and tensile strengths declined at 5 phr, likely due to nanoparticle agglomeration leading to poor dispersion, crack initiation, and stress concentration.

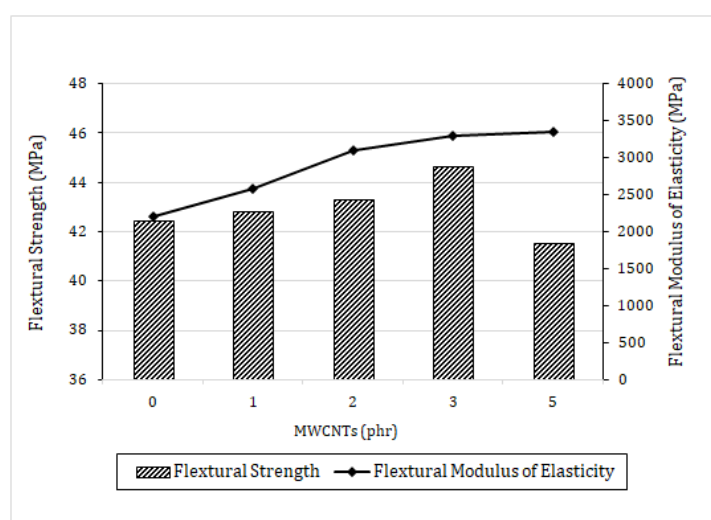


Figure 1. Flexural strength and modulus of PP/reed flour nanocomposites as a function of MWCNTs content

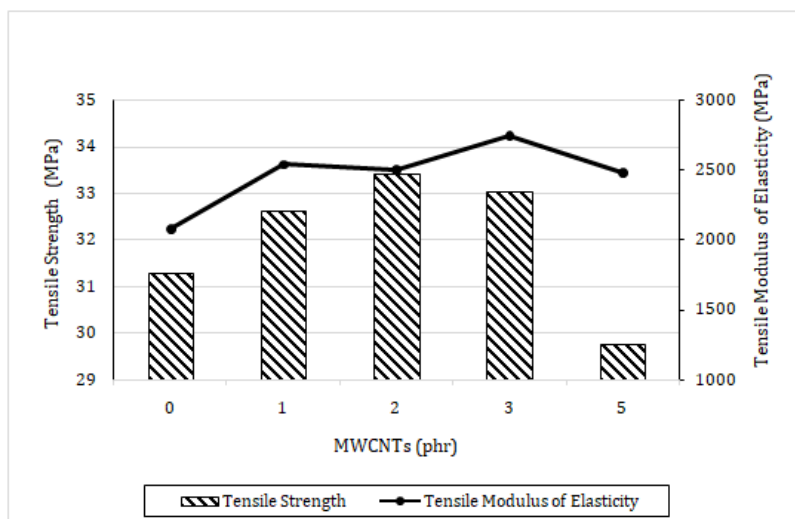


Figure 2. Tensile strength and modulus of PP/reed flour nanocomposites as a function of MWCNTs content

The results indicate that the introduction of MWCNTs increased both flexural and tensile strength. An increase in mechanical properties reveals that the stress was successfully transferred from the PP matrix to the nanostructures reinforced nanocomposite. The nanocomposites incorporating 3 phr MWCNTs exhibited the highest flexural strength and modulus of elasticity. Statistical analysis showed that the mechanical properties in terms of flexural strength and modulus were significantly influenced by the addition of MWCNTs. These results are in compliance with other researches in which greater flexural strength was achieved by applying carbon nanotubes in the PP composites (Li et al., 2007; Musso et al., 2009; Kordkheili et al., 2012). The results also verified that MWCNTs increases the tensile strength of nanocomposites but the optimum values of tensile strength and modulus correspond to the 2 phr MWCNTs specimens. However, increasing MWCNTs nanostructure adversely affects the tensile strength and elasticity modulus.

On the other hand, the addition of 5 phr MWCNTs decreased both flexural and tensile strength of nanocomposites by 7% and 10%, adversely affecting the flexural and tensile modulus of elasticity. This might be related to the ineffective dispersion of MWCNTs and nanostructure agglomerates, leading to the initiation of cracks at stress concentration points and, consequently, a decline in tensile strength.

3.2 Impact resistance

It was found that the impact strength shows the strength of the material against breakage and initiation of crack formation at the weakest regions through the microstructure of the composite, which is the connecting point between lignocellulosic material and polymer matrix (Farsi and Sani, 2014). Figure 3 shows the values of notched impact strength of the studied nanocomposites.

Impact strength, which represents resistance to fracture under sudden loading, is highly dependent on the interfacial zones within the composite microstructure. As shown in Figure 3, the inclusion of 2 phr MWCNTs enhanced impact strength by ~10%, acting as crack arresters through a bridging mechanism (Makar et al., 2005; Tavasoli Farsheh et al., 2011). However, beyond this concentration, impact resistance dropped, likely due to MWCNT clustering, which weakened the matrix's ability to transfer stress effectively (Eitan et al., 2003).

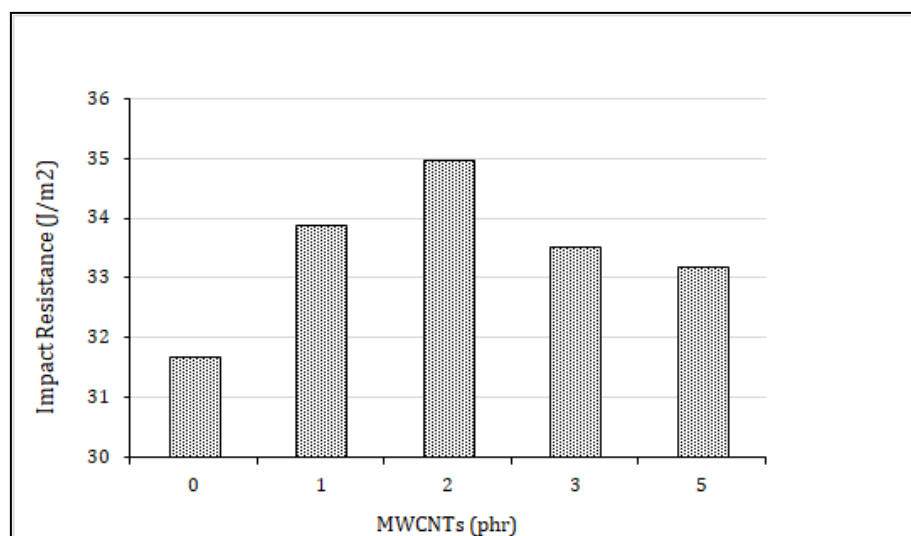


Figure 3. Impact resistance of PP/reed flour nanocomposites as a function of MWCNTs content

3.3 Water absorption and thickness swelling

Natural fibers absorb moisture due to their high hydroxyl group content, resulting in dimensional instability. Water may also permeate through capillary pathways formed at weak fiber-polymer interfaces (Espert et al., 2004). As shown in Figure 4, samples with 1 and 2 phr MWCNTs exhibited higher water absorption, while the lowest was found in 5 phr composites. Thickness swelling followed a similar trend. These findings align with prior studies highlighting the role of nanoparticle concentration on hydrophobic behavior (Abdelmouleh et al., 2007; Dhakal et al., 2007; Shinoj et al., 2010). The influence of MWCNTs addition on the water absorption after 24 h water immersion and the thickness swell of PP/RF nanocomposites are shown in Figure 4.

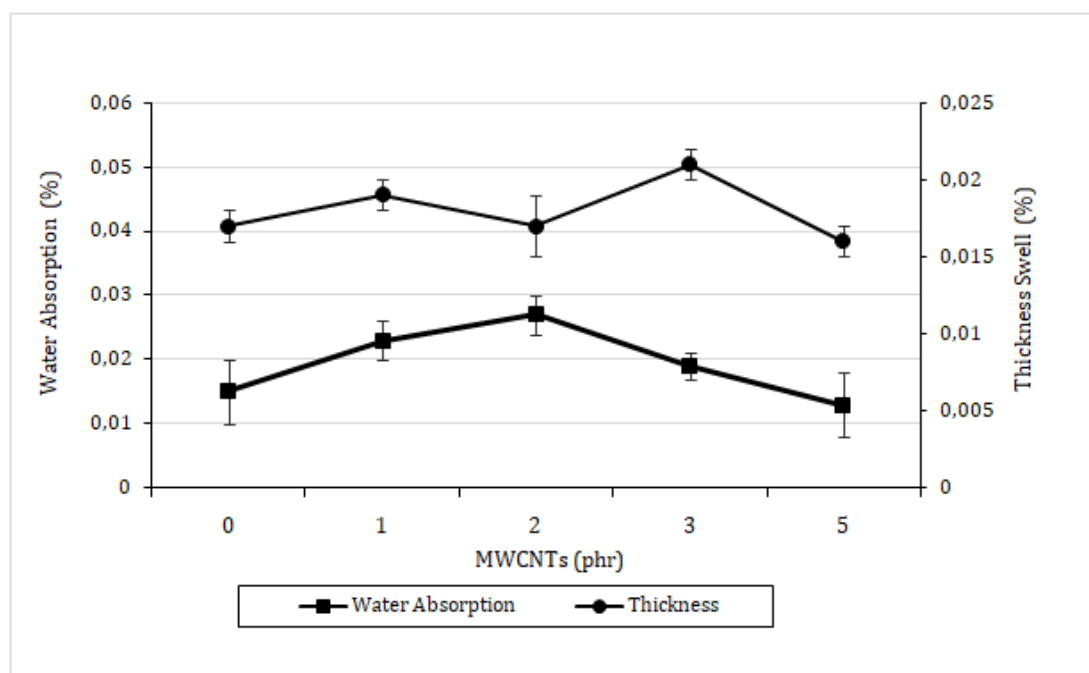


Figure 4. Water absorption and thickness swell percentage (24 hour) of PP/reed flour nanocomposites as a function of MWCNTs content

The highest water absorption values were observed in 1 and 2 phr MWCNTs incorporated composites, whereas the lowest one was found in the nanocomposites containing the highest amount of MWCNTs (5 phr). The lowest thickness swell was obtained from the experiment performed on the 5 phr MWCNTs nanocomposites.

3.4 Morphology of hybrid nanocomposites

SEM analysis (Figure 5) illustrated improved filler-matrix interaction upon MWCNT addition, with crack bridging mechanisms evident. Increased MWCNT levels, however, led to visible agglomerates, particularly at 5 phr, which created stress concentration zones prone to failure (Seshadri and Saigal, 2007).

Figure 5 shows FE-SEM micrographs showing the fracture surfaces of PP/reed flour nanocomposites containing MWCNTs at 1, 2, 3, and 5 phr.

Introducing MWCNTs to the PP/reed flour composite improved the interface between the fibers and matrix. Moreover, CNT nanostructures can act as barriers in the matrix against the cracks opening during their growth, through a crack bridging mechanism illustrated in Figure 6.

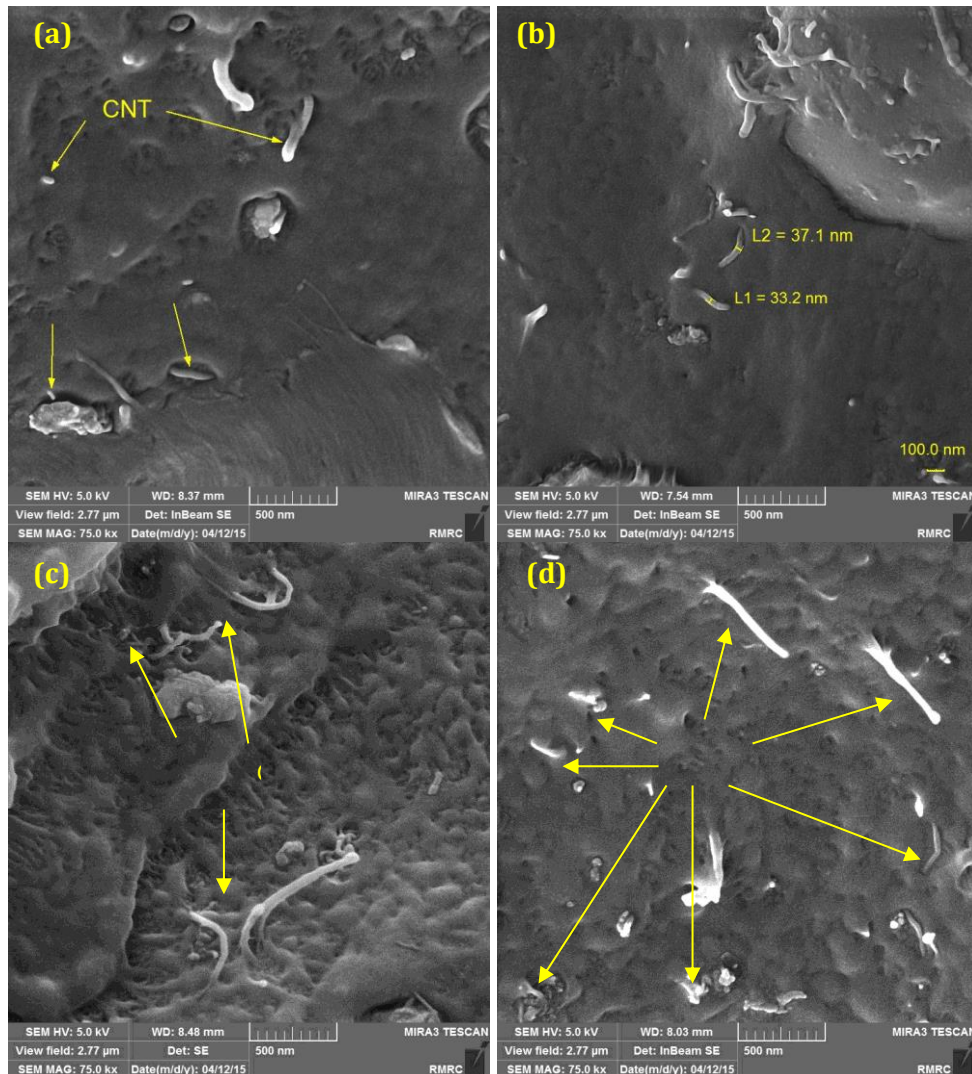


Figure 5. FE-SEM micrographs of PP/RF/MWCNT nanocomposites microstructure: a) 1 phr MWCNTs, b) 2 phr MWCNTs, c) 3 phr MWCNTs, d) 5 phr MWCNTs

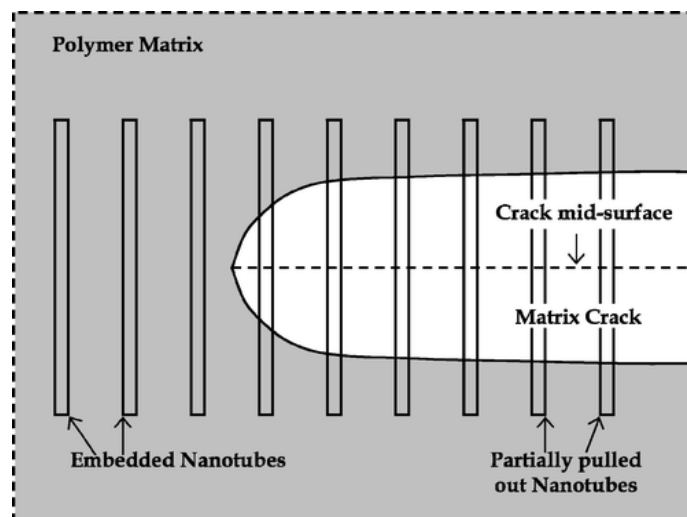


Figure 6. Schematic of crack bridging mechanism by CNTs, Reprinted from Ref. (Seshadri and Saigal, 2007)

As a result of such phenomena, a higher level of energy is needed for the crack to grow and propagate (Seshadri and Saigal, 2007). The crack bridging process is idealized as normal MWCNTs pull out from the polymer matrix, which is shown in the fracture surface of nanocomposites with all amounts of MWCNTs.

It can also be observed that some regions of the agglomerated nanoparticles can be identified from the SEM images as the MWCNTs content was increased from 3 to 5 phr, (Figures 5 (c) and (d)). The decline in the flexural and tensile strength and the impact strength value for nanocomposites incorporating MWCNTs more than 3 phr might be related to the increase in probability for nanostructures agglomeration that creates regions of stress concentrations requiring less energy to elongate the crack propagation.

4 Conclusions

- This study investigated the influence of multi-walled carbon nanotubes (MWCNTs) on the physical and mechanical properties of polypropylene/reed flour (PP/RF) nanocomposites. The results confirmed that the incorporation of MWCNTs up to 3 phr significantly enhanced the mechanical properties, particularly flexural and tensile strength. Notably, 3 phr MWCNTs led to the highest flexural performance, whereas tensile properties peaked at 2 phr.
- Despite the mechanical benefits, increasing MWCNT concentration to 5 phr resulted in performance deterioration due to nanoparticle agglomeration and poor dispersion. Furthermore, 2 phr MWCNTs exhibited optimal impact strength, while the lowest water absorption and thickness swelling occurred in the 5 phr formulation, indicating improved hydrophobicity at higher MWCNT content.
- Overall, the findings suggest that the addition of MWCNTs, particularly at 2-3 phr, is effective in reinforcing PP/reed flour composites, optimizing their structural and moisture resistance characteristics for potential industrial applications.

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Author Contributions

Seyyed Khalil HosseiniHashemi: Creating the research idea, writing the article, performing the statistical operations, and interpretation of data **Maryam Jamshidi:** Conducting the laboratory work, taking the measurement data, **Behzad Kord:** Collaboration in research idea and analysis of data, **Eshmaiel Ganjian:** writing and improving the article, **Ahmad Ehsani:** writing the article and drawing the graphs, **Nadir Ayrlimis:** writing and improving the article.

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Conflict of interest

We confirm that there is no conflict of interest.

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