Original Research Article

Improving the Functional Properties of Chitosan-Curcumin Films Using Microwave

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ABSTRACT

Recently, bio-based polymers have been utilized in packaging film production as a sustainable alternative to non-renewable synthetic plastics. This study aimed to evaluate the effects of microwaves on the functional properties of chitosan incorporated with curcumin (CSCur), and to determine the optimal microwave duration to enhance these properties. The films were subjected to microwave treatment for 30, 60, and 120 s at a constant power of 800 W and were analyzed for thickness, color/opacity, water solubility, mechanical strength, vibration, crystallinity, and surface morphology and compared to untreated samples. The results indicated that the microwave treatment did not significantly affect the thickness (ANOVA, $p>0.05$). However, the 120-second treatment significantly improved color (ANOVA, $p<0.05$) (L*: 30.70, a*: 1.91, b*: 8.93), opacity (2.19), reduced water solubility (47.51% unheated, 59.96% heated), and increased crystallinity (17.35%) compared to untreated and shorter treatment durations. Conversely, the highest tensile strength (1.30 MPa) was observed at 60 s because strength reduction occurred beyond this duration. FESEM analyses revealed that the microwave-treated CSCur films had smoother and denser surfaces with fewer pores. The changes detected in ATR-FTIR and XRD supported the observed improvements in mechanical and barrier properties, indicating rigidification of the hydrophilic domains at the OH/NH moiety and fluidization of the hydrophobic domains at the CH moiety, along with a significant increase in the crystallinity index (ANOVA, $p<0.05$). Thus, 120 s of microwave treatment is recommended to optimize the chitosan films incorporated with curcumin.

Keywords: bio-based polymers, chitosan-curcumin films, microwave treatment, packaging materials, mechanical properties, crystallinity.

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1. Introduction

Plastic pollution poses a significant environmental threat, affecting ecosystems globally (1). Due to plastic's non-biodegradable and non-recyclable nature, substantial waste is produced, with 85% ending up in landfills and oceans, harming soil quality, marine life, and biodiversity (2). Consequently, many regions are reducing single-use plastic food packaging to protect the environment and cut pollution management costs. Bio-based polymers are being researched as alternatives to synthetic plastics, offering renewable solutions (3). Natural materials like pectin, chitosan, and cellulose are biodegradable and non-toxic, providing effective food preservation films or coatings that reduce oxidation, prevent moisture loss, and inhibit microbial growth (4).

Chitosan, a chitin derivative, is commercially produced through deacetylation. It demonstrates excellent film-forming properties due to its water-holding capacity, intrinsic antimicrobial activity, and controlled release of active ingredients (5). Xie *et al.* (2021) (6) concur that chitosan is suitable for food packaging because of its biodegradability, bacteriostatic properties, and non-toxicity. However, its application in the food industry is limited by low mechanical strength and poor water barrier properties, necessitating improvements in its hydrophilicity and mechanical properties. The incorporation of bioactive compounds such as curcumin is a widely employed method for enhancing film properties. Curcumin, a natural polyphenol derived from *Curcuma longa L.*, is frequently utilized as a food colorant and spice. The integration of curcumin into functional films confers antioxidant and antimicrobial properties, rendering it suitable for food packaging applications. Dong *et al.* (2023) (7) observed that numerous studies have developed biopolymer-based films incorporating curcumin, effectively improving the quality and safety of packaged food products due to curcumin's ability to form intermolecular hydrogen bonds, which enhance the mechanical and barrier properties of films. In a recent study, Xie *et al.* (2021) (6) investigated pectin/chitosan film containing curcumin, demonstrating excellent barrier and antioxidant properties through improved polymer interactions.

Microwave technology employs electromagnetic waves with frequencies between 300 MHz and 300 GHz, heating materials by causing molecular vibrations, particularly in water, to generate heat through friction. This method offers rapid, uniform heating, energy efficiency, and precise control, making it useful for modifying packaging materials' properties. In polymers and films, microwave treatment can alter mechanical strength, crystallinity, and barrier characteristics by changing molecular structures and interactions. Chen *et al.* (2023) (8) observed that microwave treatment enhances edible film properties by breaking hydrogen bonds and increasing intermolecular crosslinking (9), thus improving mechanical properties like tensile strength (TS) and elongation at break (EAB), as well as haze and gloss values compared to other drying methods (10). Moreover, it reduces processing time and boosts energy efficiency in edible film production (11). Despite these benefits, there is limited research on using microwave treatment to improve chitosan-curcumin films. Thus, the aim of this research is to evaluate the potential of microwave treatment in enhancing functional properties of chitosan-curcumin films for food packaging applications, addressing the existing gap on microwave-induced modifications in polymer-curcumin interactions to improve performance.

2. Materials and Method

2.1 Materials

Curcumin (E-100) extracted from turmeric (Oterra Malaysia), chitosan with 78% degree of deacetylation and 52433 Da molecular weight (Sigma-Aldrich, Malaysia), food-grade beeswax (Future Food Sdn Bhd, Germany), glycerol, and acetic acid (R&M Chemicals, Malaysia) were used for the preparation of film-forming solutions.

2.2 Film formation

The process of preparing a chitosan-curcumin film was adapted from the methodology outlined by Wang *et al.* (2021) (12), with minor adjustments. Specifically, 2.0 grams of chitosan was dissolved in 100 mL of 2% acetic acid solution, while 0.2 grams of beeswax were dissolved in 2 mL of glycerol. The dissolved beeswax was added to the chitosan solution, followed by 0.1 grams of curcumin. The mixture was continuously stirred for 20 min to create a homogeneous film-forming dispersion. The film stock solution was diluted to 0.2 milligrams per milliliter with the dilution solution. Next, 25 g of the film solution was placed onto a 9-centimeter diameter glass plate and dried in a 40°C oven for 48 h to allow for evaporation of the solvent and formation of the composite film. Finally, the dried film was peeled off and stored in a desiccator away from the light.

2.3 Microwave processing

The microwave heating process was based on the method outlined by Jacquot *et al.* (2014) (9). Specifically, the edible films (stored for 24 h after drying at ambient conditions (25°C ± 1°C, RH 50% ± 5%)) were set up individually in the microwave device and heated for different periods (0, 30, 60, and 120 s) at a power of 800 watts. To prevent vapor production and diffusion into the microwave oven, 300 mL of distilled water was placed in a beaker alongside the edible films in the microwave device during the heating process.

2.4 Characterization of chitosan film loaded with curcumin

Film thickness

The thickness of the film was measured according to Nazir and Wani (2022) (13). A digital Vernier caliper (Mitutoyo 500-196-30, Japan) was used, and the film thickness (dimension of 6 × 1 cm) was measured at five random points. The mean value of the thickness of the film was then expressed in millimeters (mm) and used to calculate the mechanical and optical barrier properties. Triplicates were conducted and the results were averaged.

Colour and optical properties

The colour of the film samples was measured using a CR-400 chromameter (Konica Minolta, Japan). Prior to the analysis, the chromameter was calibrated using standard black and white tiles. Xenon flash lamp was the light source of the chromameter. To measure the color of the film, it was placed on a Petri dish, and the temperature of the film was found to be similar to the ambient temperature (20±1 °C). The results were recorded as L*, a*, and b* values, which represent the luminosity, red/green, and yellow/blue colour parameters of the film, respectively. A standard white plate was used as the background. The analysis was performed three times, and the results were recorded on average.

The opacity of the films was determined using the method described by Sun *et al.* (2020) (14). The edible film was cut into a rectangular shape (1 × 5 cm^2) and placed in a cuvette. Absorbance was measured at 600 nm

using an empty cuvette as a reference. The analysis was carried out for three times and the results were recorded in average. Opacity (Equation 1) was calculated using the following formula:

Opacity = A600/t (1)

where t is thickness

Water solubility

Prior to the analysis, the films were cut into 2×2 cm pieces, and the initial weight of the film was measured (Wi = Initial dry weight). The films were then immersed in heated at 100 °C (to simulate extreme thermal conditions relevant for high-temperature food packaging applications) and unheated deionized water for 15 min (selected as sufficient time to evaluate film solubilization behavior without total disintegration). Samples were removed and dried at 40 °C in a drying oven for 24 h. Once the films were dried, their weight was measured (Wf = Final dry weight). The water solubility of the films was calculated as follows (Equation 2):

Water solubility % =
(W_i-W_f)/W_i ×100 (2)

Mechanical properties

The analysis of the mechanical properties of the films was slightly altered by referring to Wang *et al.* (2019) (15). The films were cut into 6 × 1 cm pieces, the test speed was 10 mm/s, and the initial clamping distance was 200 mm. Tensile strength (MPa), elongation at break (%), and Young's modulus (MPa) were measured using a texture analyzer (TA. XT plus, Stable Micro System company, Surrey, UK). The analysis was performed three times, and the results were recorded on average. The mechanical properties of the chitosan-curcumin films were calculated using the Equations (3), (4) and (5)

Tensile strength (TS)= (Maximum load at breaks in N (F_max)) / (Film cross-sectional area in ⟦mm⟧^2 (S)) (3)

Elongation at break (EAB)= (L_1-L_0)/L_0 ×100 (4)

where, L0 was the initial length of the film (mm) and L1 referred to the length of film after stretching (mm)

Young^' s modulus (YM)= TS/EAB (5)

2.5 Field emission scanning electron microscopy (SEM) analysis

The morphology or surface of the film was evaluated using Scanning Electron Microscopy (FESEM, Hitachi SU8010, Japan). Before running the analysis, the films (2×2 mm) were vertically adhered to copper stubs with their surfaces. The film was then sputtered with a thin layer of Au. SEM images were collected at 5.0 kV accelerating voltage and 500× magnification was used to view the film samples (14).

2.6 X-ray diffraction (XRD)

The XRD patterns of the films were measured using an X-ray diffractometer (Shimadzu XRD-6000, Japan) with a Cu-Kα radiation source (λ = 1.54 Å) operated at 40 kV and 100 mA. The scanning range of 2θ (diffraction angle) was 3-120° and the scanning speed was 3°/min. The results were analyzed using Origin software (16). Analyses were conducted on three independent replicates.

2.7 Attenuated Total Reflectance (ATR)

The ATR spectra of the composite films were determined using an FTIR spectrophotometer with ATR accessories (Nicolet550 FT-IR Spectrometer, Thermo Nicolet Corporation, USA). The measurements were performed

using a measurement probe directly in contact with the surface of the films, and the results were recorded in a wavenumber ranging of 4000-500 cm^{-1} at a spectral resolution of 16 cm^{-1} with 16 scans. Analyses were conducted on three independent replicates.

2.8 Statistical analysis

Statistical analyses were performed using the method described by Wang *et al.* (2023) (17). The tests were repeated thrice, and the average results were reported. The mean difference was analyzed using the commercial statistical software SPSS23 (SPSS, Inc., Chicago, IL, USA). All data were analyzed using one-way analysis of variance (ANOVA). The results are presented as mean values and standard deviations (SD), and Duncan's test was performed to compare differences in mean values, with $p < 0.05$, indicating a significant difference. All statistical data were assessed as the mean value ± standard deviation of the film.

3. Results and Discussion

3.1 Thickness and water solubility

Table 1 presents data on film thickness and water solubility at various microwave heating durations. The microwave treatment did not significantly impact film thickness, as no notable differences were observed among the samples (ANOVA, $p>0.05$). This finding aligns with previous study by Jacquot *et al.* (2014) (9), although film compositions and microwave parameters differed slightly in these studies. Solubility critically affects the functionality of edible films, especially in preserving high-water activity or perishable foods, where low solubility is crucial (18). Conversely, water solubility is advantageous for food coating and encapsulation (19). This study assessed solubility under unheated and heated conditions, revealing that extended microwave heating significantly reduced (ANOVA, $p<0.05$) water solubility (Table 1). The reduction is due to microwave treatment enhancing the compactness and molecular aggregation of the chitosan-curcumin film through increased intermolecular interactions, primarily hydrogen bonding between chitosan and curcumin (20), resulting in a denser composite film with fewer voids for water molecules (21).

Similar findings were reported by earlier studies that developed a microwave-treated film blending sodium alginate and Na-CMC. This film exhibited enhanced resistance to water penetration due to the formation of a tightly cross-linked structure between the polar functional groups of sodium alginate and Na-CMC (22). In the current study, CSCur-MW120 demonstrated the lowest water solubility under both heated and unheated conditions, showing a statistically significant difference from other samples (ANOVA, $p<0.05$). This observation suggests that CSCur-MW120 possessed the highest hydrophobicity, which can be attributed to its high crystallinity index, as illustrated in Fig. 2. The increased crystallinity resulted in reduced film dissolution and solubility in water (23). Notably, the film's thickness did not significantly affect its solubility (ANOVA, $p>0.05$).

Table 1. Thickness and water solubility of film at different microwave heating time

Sample	Thickness (mm)	Water solubility (%)	
		Unheated	Heated
CSCur-MW0	0.126 ± 0.008^{NS}	58.58 ± 0.74^{a}	63.41 ± 1.51^{a}
CSCur-MW30	0.124 ± 0.006	56.95 ± 0.46^{a}	62.60 ± 1.31^{ab}
CSCur-MW60	0.124 ± 0.004	54.23 ± 0.77^{b}	62.19 ± 0.32^{ab}
CSCur-MW120	0.126 ± 0.007	47.51 ± 1.98^{c}	59.96 ± 0.20^{b}

Note: Data were presented in mean ± standard deviation (n=3). Mean values with different superscript letter in the same column had significant different ($p < 0.05$), meanwhile ^{NS} indicated no significant different.

3.2 Color analysis and optical properties

The visual attributes of a film, such as color and opacity, are pivotal in food packaging development, influencing consumer acceptance and extending shelf-life by preventing discoloration and nutrient loss (24). Table 2 presents color parameter measurements and opacity of chitosan-curcumin film influenced by different microwave heating time (0, 30, 60, and 120 s). The results show that increased microwave heating duration significantly reduced film luminosity (ANOVA, $p<0.05$). This supports Jacquot *et al.* (2014) (9), who found extended microwave treatment darkened chitosan films, reducing L* values. Longer heating also significantly increased a* values (ANOVA, $p<0.05$), indicating a reddish tint, while positive b* values were due to yellow curcuminoid pigments in curcumin. Prolonged microwave exposure increased the yellow coloration, raising b* values. Curcumin in chitosan films enables UV light absorption through its phenolic groups, as studied by Kaya *et al.* (2023) (25). Table 2 shows that film opacity significantly increased with longer microwave heating (ANOVA, $p<0.05$), with CSCur-MW120 opacity at 2.19 ± 0.10, almost double that of CSCur-MW0, indicating negative effects of microwave heating on opacity. The lowest L* value for CSCur-MW120 (Table 2) confirms it was the darkest film. These results align with Sun *et al.* (2020) (14), who noted that lower L* values in bio nanocomposite films corresponded to higher opacity.

3.3 Mechanical properties

Key textural parameters for films, such as tensile strength, elongation at break, and Young's modulus, are crucial for assessing film integrity and stress resistance (20). The impact of microwave duration on the mechanical properties of chitosan-curcumin film is shown in Table 3. Control films exhibited tensile strength, elongation at break, and Young's modulus of 0.36 MPa, 39.56%, and 0.92 MPa, respectively. As microwave treatment duration increased, tensile strength of CSCur films rose significantly ($p<0.05$), peaking at 1.30 MPa after 60 seconds. This increase in tensile strength is attributed to higher cross-linking density, which decreases elongation percentage, resulting in less elastic films (22). Consequently, elongation at break diminished with longer microwave periods. However, prolonged microwave exposure can affect the film structure, causing a substantial drop in tensile strength from 1.30 to 1.04 MPa in CSCur-MW120 films. Similar degradation was reported by Ullah *et al.* (2021) (21), where Na-CMC/Eudragit L-100 film strength declined after 120 seconds of microwaving, disrupting cellulose and polymer chain integrity.

Table 2. Luminosity (*L**), chromatic coordinates (*a** and *b**) and opacity of chitosan-curcumin film influenced by different microwave heating time.

Sample	Appearance	L*	a*	b*	Opacity
CSCur-MW0		38.35 ± 0.48^{a}	0.42 ± 0.03^{d}	6.46 ± 0.28^{c}	1.21 ± 0.04^{d}
CSCur-MW30		34.57 ± 0.22^{b}	0.52 ± 0.02^{c}	7.19 ± 0.24^{c}	1.67 ± 0.05^{c}
CSCur-MW60		32.32 ± 1.68^{b}	0.75 ± 0.04^{b}	7.94 ± 0.68^{b}	1.97 ± 0.16^{b}
CSCur-MW120		30.70 ± 0.28^{c}	1.91 ± 0.06^{a}	8.93 ± 0.18^{a}	2.19 ± 0.10^{a}

Note: Data were presented in mean ± standard deviation (n=3). Mean values with different superscript letter in the same column had significant different ($p < 0.05$).

Microwave duration also significantly affects Young's modulus; increased crystallinity from extended microwave treatment enhances both Young's modulus and tensile strength (26). This is due to the formation of hydrogen bonds, resulting in a more compact and rigid film structure, as investigated by Uygun *et al.* (2020) (27). Thus, microwave treatment improves film mechanical properties by enhancing polymer blend integration through increased intermolecular forces, altering the polymer molecular structure (28).

3.4 Morphology

The cross-sectional morphology of the film samples (Fig. 1) reveals that the untreated film (CSCur-MW0) had a rough, bumpy surface, consistent with previous finding (29). Microwave-treated films displayed smoother surfaces with fewer pores. Cross-sectional analysis showed significant differences; microwave-treated films had smoother textures, more compact polymer chain arrangements, and denser cross-sections without visible pinholes. This was due to microwave treatment enhancing hydrogen bonding between chitosan and curcumin, improving matrix dispersion, as noted by Wang *et al.* (2014) (30). Stronger intermolecular forces reduced film holes, enhancing mechanical properties (29), consistent with mechanical analysis results showing improved tensile strength and Young's modulus.

Table 3. The mechanical properties of the film at different microwave heating time.

Sample	Tensile strength (MPa)	Elongation at break (%)	Young's Modulus (MPa)
CSCur-MW0	0.35 ± 0.02^{d}	39.56 ± 0.83^{a}	0.92 ± 0.07^{d}
CSCur-MW30	0.56 ± 0.03^{c}	38.49 ± 0.53^{a}	1.46 ± 0.06^{c}
CSCur-MW60	1.30 ± 0.05^{a}	26.24 ± 0.18^{b}	4.94 ± 0.14^{b}
CSCur-MW120	1.04 ± 0.01^{b}	19.80 ± 0.47^{c}	5.26 ± 0.06^{a}

Note: Data were presented in mean ± standard deviation (n=3). Mean values with different superscript letter in the same column had significant different ($p < 0.05$).

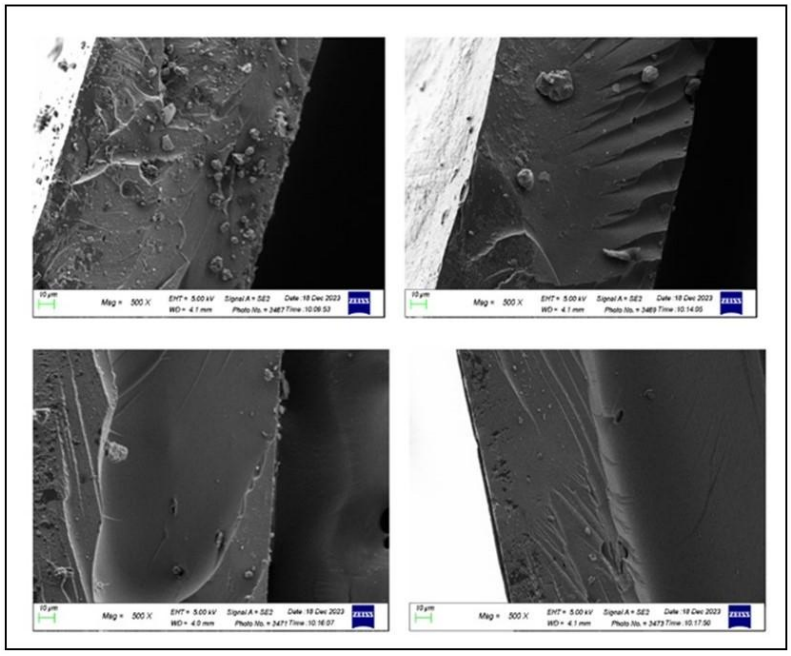

Figure 1. Cross-sections of (a) CSCur-MW0, (b) CSCur-MW30, (c) CSCur-MW60 and (d) CSCur-MW120 at magnification of 500x.

3.5 X-ray diffraction

X-ray diffraction (XRD) analysis assessed the impact of microwave heating duration on the Cs-Cur film's crystalline structure, as shown in Fig. 2. Pure chitosan had a crystallinity index of 9.73 ± 0.15%, consistent with Tang *et al.* (2022) (31), identified chitosan primary peak at $2\theta = 19.8$, indicating amorphous nature. Beeswax exhibited a sharp peak, characteristic of its orthorhombic structure (32). When blended with chitosan, beeswax peaks diminished, indicating a semi-crystalline film formation (24). Microwave treatment increased the diffraction peak, with the crystallinity index

of CSCur films rising significantly ($p < 0.05$) after 120 seconds. This increase is due to enhanced intermolecular interactions from microwave treatment (20). Liu *et al.* (2023) (33) noted that high crystallinity indicates a stable and ordered molecular structure, resulting in a more compact film with improved barrier properties due to the inert and impermeable crystal structure (27). Therefore, a film's crystallinity affects its water solubility, with CSCur-MW120 showing the highest crystallinity index and the lowest water solubility, as water molecules traverse crystal regions more slowly (27).

3.6 Attenuated Total Reflectance-FTIR

ATR-FTIR measurements were conducted to understand molecular interactions and the impact of varying microwave heating times on chitosan-curcumin film. Fig. 3 exhibits the infrared spectra of curcumin, chitosan, CSCur-MW0, CSCur-MW30, CSCur-MW60, and CSCur-MW120 films. The characteristic peak of curcumin at 911.50 cm^{-1} (Fig. 3a), related to the benzene ring structure (34), broadened and shifted to 922.01-922.49 cm^{-1} upon inclusion in the film. This indicates the benzene ring

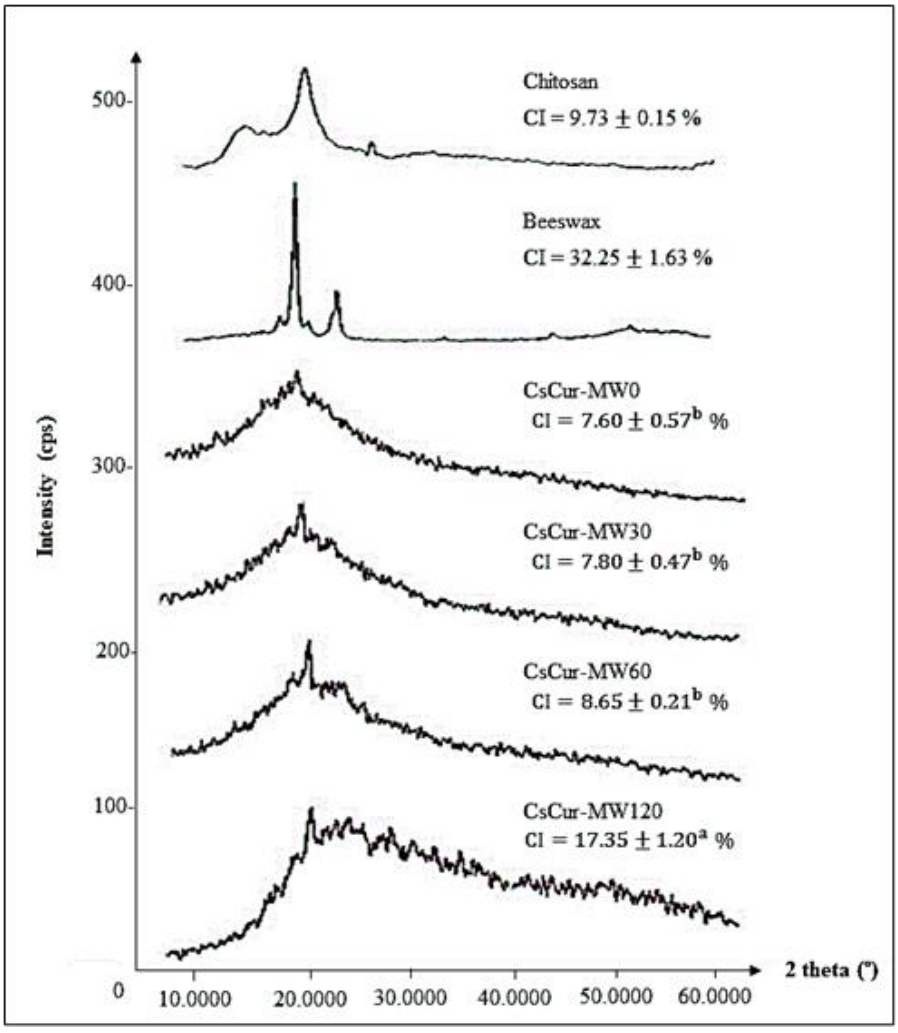

Figure 2. X-ray diffractograms of chitosan, beeswax, CSCur-MW0, CSCur-MW30, CSCur-MW60 and CSCur-MW120 treated films

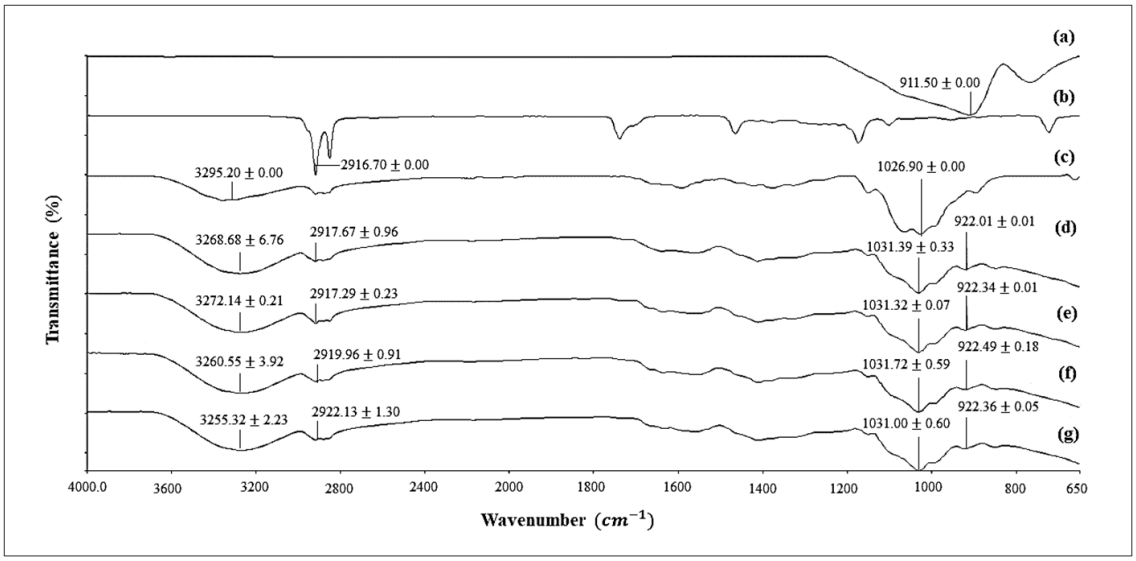

Figure 3. ATR-FTIR spectra of (a) Curcumin, (b) Beeswax, (c) Chitosan, (d) CSCur-MW0, (e) CSCur-MW30, (f) CSCur-MW60 and (g) CSCur-MW120.

structure's presence in all film samples post-microwave treatment. The hydrophobic benzene ring in curcumin reduces the film's water solubility and causes poor dispersion, leading to agglomeration and affecting the film's compact structure compared to untreated sample. Consequently, the number of free OH groups available for interaction with water is limited due to hydrophobic interactions and hydrogen bond formation. After adding beeswax to the chitosan film, the original peak at 2916.70 cm^{-1} (Fig. 3b) broadened and shifted to 2917.67 cm^{-1} (Fig. 3d), indicating an abundance of carbon-hydrogen bands in beeswax (35). The significant chitosan peak at 1026.90 cm^{-1} (Fig. 3c) attributed to C-O stretching vibration (36), remained but shifted to 1031.00-1031.72 cm^{-1} in all film samples, showing polymer compatibility during film preparation. Gao *et al.* (2023) (37) noted that intermolecular and intramolecular hydrogen bonding could cause such wavenumber shifts.

Compared to the microwave-treated film samples, the OH/NH wave number decreased significantly from 3268.68 to 3255.32 cm^{-1}, indicating increased rigidification of the polymer matrix. Ullah *et al.* (2021) (21) observed a similar reduction in the OH/NH wave number in Na-CMC/Eudragit L100 composite films treated with microwaves for longer periods, attributed to rapid moisture loss and new bond formation (hydrogen, electrostatic, and van der Waals) between Na-CMC and Eudragit L100 chains. This suggests more bonding, such as hydrogen bonds, in the CSCur film, leading to a more compact structure with increased microwave exposure (27). Concurrently, the rise in the wavenumber of the hydrophobic asymmetric CH band (2920-2930 cm^{-1}) from 2917.67 to 2922.13 cm^{-1} indicates fluidization of the hydrophobic domain due to absorbed water removal during microwave heating (38). Ullah *et al.* (2021) (21) also noted that microwaves could rearrange polymer hydrophobic moieties, leading to increased tensile strength and decreased water solubility with extended microwave periods. Mahmood *et al.* (2023) (22) confirmed that increased hydrophobic moieties in Na-CMC

films microwaved for longer durations enhanced the composite film's mechanical strength.

4. Conclusion

This study demonstrated the microwave treatment significantly improved several properties of the CSCur films without affecting thickness (ANOVA, $p>0.05$). The 120-second microwave treatment (CSCur-MW120) produced the lowest water solubility, highest opacity, and crystallinity index with smooth and dense surface (ANOVA, $p<0.05$). Mechanical properties peaked at 60 seconds of treatment, with the highest tensile strength observed; prolonged treatment beyond this led to strength reduction. Microwave treatment enhanced intermolecular interactions (primarily through hydrogen bonding, as confirmed by ATR analysis) and crystallinity (XRD results), contributing to increased rigidification of hydrophilic domains, fluidization of hydrophobic domains, and stable film structure. Overall, microwave treatment proved to be an effective method for enhancing the functional properties of CSCur films. These findings demonstrate the potential of microwave-treated chitosan-curcumin films as a sustainable and effective alternative for food packaging applications, addressing the need for environmentally friendly alternatives to synthetic plastics.

Authorship contribution statement

SMSSB: Implementation of the research, analysis of the results and writing of the manuscript. **HMN**: editing and formatting the manuscript. **NM**: conceived the original and supervised the project.

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Conflict of Interest

The authors affirm that they have no competing financial interests or personal connections that could be perceived as having impacted the work described in this article.

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