

Towards Sustainable Food Packaging: A Study of Konjac Glucomannan and κ -Carrageenan Synergy

Baptiste Lenfant, Raphaël Haumont

*ICMMO, Institut de Chimie Moléculaire et des Matériaux d'Orsay, UMR CNRS 8182,
Université Paris-Saclay, 91400, Orsay, France
E-mail: baptiste.lenfant@universite-paris-saclay.fr*

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Abstract: This study investigates the synergistic effect of konjac glucomannan (KGM) and κ -carrageenan (κ -CG) in developing sustainable food films. KGM and κ -CG were dissolved at various ratios (20:80 to 80:20 KGM/ κ -CG ratio), casted, and dried. The resulting films were characterized by their mechanical properties, water vapor permeability, and water solubility. The results showed that the combination of KGM and κ -CG resulted in a synergistic effect on the water solubility of the films, which decreased significantly compared to the individual components. The tensile strength increased significantly while the elongation at break decreased the more κ -CG was added. An optimal point was found at a 60:40 ratio. The water vapor permeability of the films was also significantly higher compared to PVC film or κ -CG film, indicating a lesser barrier property. X-ray diffraction investigation shows that films are amorphous (whereas pure kappa is crystallized), and no co-crystallization was observed based on the different KG/ κ -CG ratios. Overall, this study demonstrates the potential of combining KGM and κ -CG to develop sustainable food films with enhanced properties. Using natural polysaccharides in the food industry can contribute to developing eco-friendly and sustainable packaging solutions.

Keywords: Polysaccharides; Biodegradable films; Kappa-carrageenan; Konjac glucomannan; Synergy; Mechanical properties.

1. Introduction

Addressing the contemporary imperative to reduce plastic usage poses a multifaceted challenge, particularly evident in the food industry, where plastic packaging takes the form of stretch or rigid plastics. Such materials need to adhere to stringent criteria, such as being a sound water barrier or having suitable physical properties [1]. In response to this challenge, polysaccharides have emerged as a promising alternative to plastics. The film-forming capabilities of polysaccharides, extensively explored in the food packaging sector [2], offer a diverse array of film qualities due to the broad spectrum of available polysaccharides [3]. Additionally, biopolymers present the advantages of being biocompatible, biodegradable, and cost-effective [4]. However, the industrial application of polysaccharide films for food purposes is limited by their brittleness and hydrophilicity. Various factors, such as the nature of the food, chemical components, preparation method, packing material, and storage conditions, influence the longevity of food products [5].

Edible films can be produced from different macromolecules, including polysaccharides, proteins, and lipids [6,7]. While films made from single macromolecules like alginate, carrageenan, cellulose, chitosan, or pectin have been explored [8–11] there is also a trend towards composite films using mixed polysaccharides, combining their inherent qualities to enhance overall properties. Examples include cellulose with starch [12], konjac with xanthan gum [13], chitosan and starch [14], and kefir and starch [15].

Despite considerable interest in polysaccharide films, the film-forming potential of Konjac Glucomannan/ κ -carrageenan combinations remains understudied, with a notable exception being a prior patent from the laboratory [16]. κ -carrageenan, renowned for its robust film-forming capabilities [17–19] is a staple in the food industry due to its ability to gel and stabilize products [20].

Chen et al. demonstrated significant interactions between Konjac Glucomannan and κ -carrageenan in gels [21], laying the groundwork for our exploration of this interaction within a film framework. The main objective of this study was to combine the properties of two polysaccharides at different ratios. The aim was to use the flexibility of konjac films with the cohesiveness of κ -carrageenan without adding other elements. This allowed one to manipulate the gel quickly and efficiently to fix the breaking with its thermoreversible properties.

2. Materials and methods

2.1 Materials

Konjac Glucomannan gum (E425 (i)) (KGM) was purchased from KALYS SA (France), and (κ -CG) was supplied by Sigma-Aldrich (USA). Both reagents have high purity and are food grade.

2.2 Sample preparation

For the preparation of all films, different proportions were used by w/w, given in Table 1. Films made of one polysaccharide (κ -CG and KGM) were prepared as follows. κ -CG or KGM (2 g at 100% ratio) was dissolved in 100 g of distilled water at 75°C with continuous mechanical stirring for 30 minutes (650 rpm for the first 5 minutes and then increasing to 1250 rpm). The solution was cooled down to 50°C before film casting by pouring 90 g of the solution onto a round polystyrene casting plate (15 cm \times 0,3 cm) and dried for 12 h at 45 \pm 5°C using a food dehydrator. Hydrogels were placed at room temperature (20°C) for 24 h, becoming films, and manually peeled off from casting plates. The same method was used for the composite films, but the two polysaccharides were mixed thoroughly before dissolution. All the composite solutions have a quantity of 2 g of polysaccharides. An example of such a film is presented for a K4 blend in Table 1.

Table 1. Coding of film-forming samples.

Code	Blend
K0	κ -CG(100%)
K8	κ -CG(80:20)KGM
K6	κ -CG(60:40)KGM
K5	κ -CG(50:50)KGM
K4	κ -CG(40:60)KGM
K2	κ -CG(20:80)KGM

2.3 Characterization of Kappa-Konjac films

2.3.1 Thickness measurement

Film thickness was measured using a digital micrometer (Model MDC-25PX, Mitutoyo Co., Tokyo, Japan) to the nearest \pm 0.001 mm at 10 random positions for each film sample after 24 h at room temperature. The mean value of the 10 measurements was determined and used in the calculations for mechanical properties and water vapor permeability.

2.3.2 Film solubility in water

Water solubility (WS) was measured using the method described by Ganesan et al., 2019 [22] with slight modifications. For each dried film sample, the initial weight was measured beforehand. The sample was then immersed in 30 mL of water in a beaker at room temperature (20°C) for 1h under magnetic stirring at 200 rpm. The solution was filtered, and the undissolved film was poured into a crucible. The hydrated film was allowed to dry at 105°C in an oven for 12 h. The dried undissolved film was then weighed to obtain its final weight. The water solubility was expressed as a percentage calculated using the following equation:

$$WS (\%) = \frac{w_0 - w_1}{w_0} \quad (1)$$

Where w_0 is the initial weight of the dry film and w_1 is the final weight of the undissolved dried film.

2.3.3 Water uptake ratio

A gravimetric method was used to determine the water uptake ratio (WUR). A square sample (3 \times 3 cm) of a dried film was immersed in distilled water (30 mL) for 30 min. The hydrated film sample was then removed and weighed. The WUR was expressed as a percentage and was calculated using the following equation:

$$WUR (\%) = \frac{w_h - w_d}{w_d} \quad (2)$$

Where w_h is the weight of the hydrated film after immersion in water, and w_d is the initial weight of the dry film before immersion in water.

2.3.4 Determination of mechanical properties

The tensile strength (TS) and elongation at break (EB) of the films were determined using a texture analyzer (TA1, AMETEK Lloyd Instruments, USA) according to the ASTM D882-18 procedure [23]. The film samples were conditioned at room temperature (20°C) for 24 h. With an initial grip distance of 90 mm and a crosshead speed of 100 mm/min, and a load cell of 1 kN, four film samples were uniformly cut into strips (10 cm × 2 cm). The TS was calculated using the following equation:

$$TS(MPa) = \frac{F_{max}}{A} \quad (3)$$

Where F_{max} is the maximum load of the specimen, and A is the initial cross-sectional area. EB expressed as a percentage was calculated using the following equation:

$$EB(\%) = \frac{\Delta l}{l_0} \quad (4)$$

Where Δl is the change in the initial gauge length ($\Delta l = l_{final} - l_0$) and l_0 is the initial length of the film sample.

2.3.5 Determination of water vapor permeability

The water vapor permeability (WVP) of the films was determined using a modified technique described by Sobral et al. [24]. The film was firmly sealed on a polystyrene cup filled with silica gel. The cups were then placed in a desiccator with distilled water. The relative humidity (RH) inside the desiccator was 76%. The cups were then weighted (± 0.01 g) each 2 h until 7 points were obtained. A linear regression analysis was used to fit the weight vs. time plot data to calculate each line's slope. Water vapor permeability ($\text{g}\cdot\text{m}^{-1}\cdot\text{Pa}^{-1}\cdot\text{h}^{-1}$) was calculated using the following equation:

$$WVP = \frac{\Delta w \times x}{A \times P_{sat}^{25^\circ\text{C}} (RH_1 - RH_2)} \quad (5)$$

Where Δw is the plot ($\text{g}\cdot\text{h}^{-1}$), x is the film thickness (m), A is the exposed area of the film ($4.42\cdot 10^{-3}$ m²), $P_{sat}^{25^\circ\text{C}}$ is the vapor pressure of water at 25°C (3170 Pa), RH_1 is the relative humidity inside the desiccator (76%), and RH_2 is the relative humidity inside the cup (20%).

2.3.6 Fourier Transform Infrared Spectroscopy (FT-IR) analysis

The IR spectra were recorded with a Vertex 70 FT-IR spectrometer (Bruker, Germany) with an attenuated total reflectance (ATR) with a Germanium crystal. The spectrum was obtained in the range of 700-3700 cm^{-1} wavenumbers with a resolution of 4 cm^{-1} . A total number of 37 scans were performed at ambient temperature (22°C).

2.3.7 X-ray diffraction (XRD) analysis

The blended films were characterized by Bragg-Bentano θ -2 θ X-ray diffractometry (Panalytical X'Pert Philips) using Cu-K α radiation ($\lambda = 1.54056$ Å) in the 2 θ range from 5 to 35°.

2.4 Statistical analysis

Data obtained were analyzed for statistical tests using RStudio software (RStudio: Integrated Development for R. R Studio, Boston, MA, USA). Using Tukey's multiple range test, one-way ANOVA was used to determine the significance at $p < 0.05$. The results obtained were expressed as mean \pm standard deviation. All the tests were carried out in triplicates or more.

3. Results and discussion

3.1 Film appearance and thickness

All the composite films exhibited uniform and bubble-free surfaces, displaying flexibility and transparency. Easy peeling off from casting plates further emphasized the practicality of these films. The thickness variation of the composite films is detailed in Figure 1, presented as a binary phase diagram with κ -carrageenan concentration. Here, 0% κ -carrageenan corresponds to 100% Konjac Glucomannan (2 g in 100 mL of water, equating to 50% in the Figure representing an equal mix of κ -CG and KGM). The findings indicated a significant increase in film

thickness (p -value < 0.05) compared to films produced at κ -CG concentrations below 50%. The observed inflection point could be attributed to intricate interactions within an interpenetrating network involving κ -CG and Konjac chains, a phenomenon akin to the κ -CG-KGM gels described by [21], potentially contributing to the augmented film thickness. The film thickness ranged from 150 to 32 μm , revealing distinct trends. Beyond the 50% concentration, a change in slope indicated a transformation within the network structure.

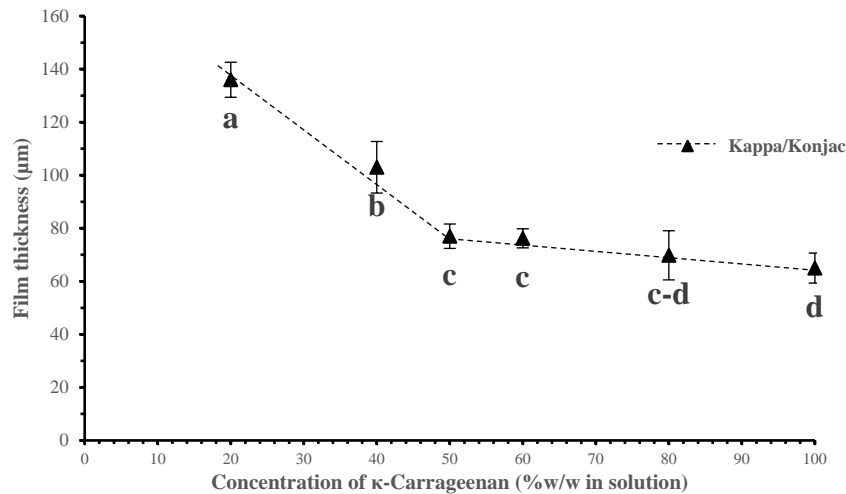


Figure 1. Thickness of composite film with different concentrations of κ -carrageenan (20% to 100%). a–d Values with different letters are significantly different ($p < 0.05$). Error bars show standard deviation.

3.2 Water solubility

A significant decrease in water solubility was found at all ratios of composite films (Figure 2.A). The monolayered films with 100% KGM or κ -CG (K0 and K10) have the highest solubility of $32 \pm 1.7\%$ and $39 \pm 1.6\%$, respectively. At this composition, all the chains in both polysaccharides can interact easily with water, as the monophasic-biopolymer had more sites to attach with water. A decrease in solubility when both the polysaccharides are mixed could be explained by the interactions between the KGM chains and the κ -CG chains, forming an interpenetrating network [21], diminishing the interactions with water. With K8, a decrease of 10% in solubility is observed. At K5, a significant increase in water solubility appears compared to the adjacent concentrations. All the composite films generally have a solubility in water under 40% (w/w) at 20°C over 1h, suggesting that films were partially soluble in water. The water solubility of composite films produced in the present study is comparable to the edible films produced by other authors, such as chitosan films [25], konjac films [26], or protein films [27].

3.3 Water uptake ratio

The water uptake ratio shows the swelling of the films when in contact with water, as shown in Figure 2.B. No significant variation was found in the initial weights of the samples. The trend was the same after the water uptake for the composite films; the water uptake ratio was constant (around 2000% of the initial weight of the film samples). However, the same experiment was done with monolayer-biopolymer (100% of κ -CG and KGM). When entering a concentration of 10 g.L^{-1} (1% in solution), both Konjac Glucomannan and κ -carrageenan showed a significant increase ($p < 0.05$), while in the composite film, the WUR stayed the same. These results show interactions inside the network, keeping the film from swelling, which can be linked with the decrease in water solubility. Swelling still occurs, as shown in Figure 3.B and C., before and after water immersion, and the thickness of the K4 film increases from $103 \pm 9 \mu\text{m}$ to $2211 \pm 105 \mu\text{m}$.

3.4 Water vapor permeability

The best mechanical and water solubility properties of composite films are seen in K4, K5, and K6 films. The water vapor permeability (WVP) of these films was tested. The same tests were made for monolayered κ -carrageenan films and a commercial PVC film (control), and the results are shown in Table 2. A significant increase in WVP was found in composite films. The more KGM was added to the film, the less the film stopped the water. This increase could be attributed to the hydrophilic nature of KGM. As the film is thicker when adding KGM, the more residual water can act as a plasticifier, leading to structural modification of the polymer network, becoming less dense and thus a weaker barrier to water. Water acting like a plasticizer affects the reorganization of the polysaccharide network and increases the free volume, allowing water molecules to diffuse more efficiently,

leading to an increase in the WVP. It must be noted that the same order of magnitude is observed in the composite film, monolayer film, and control film.

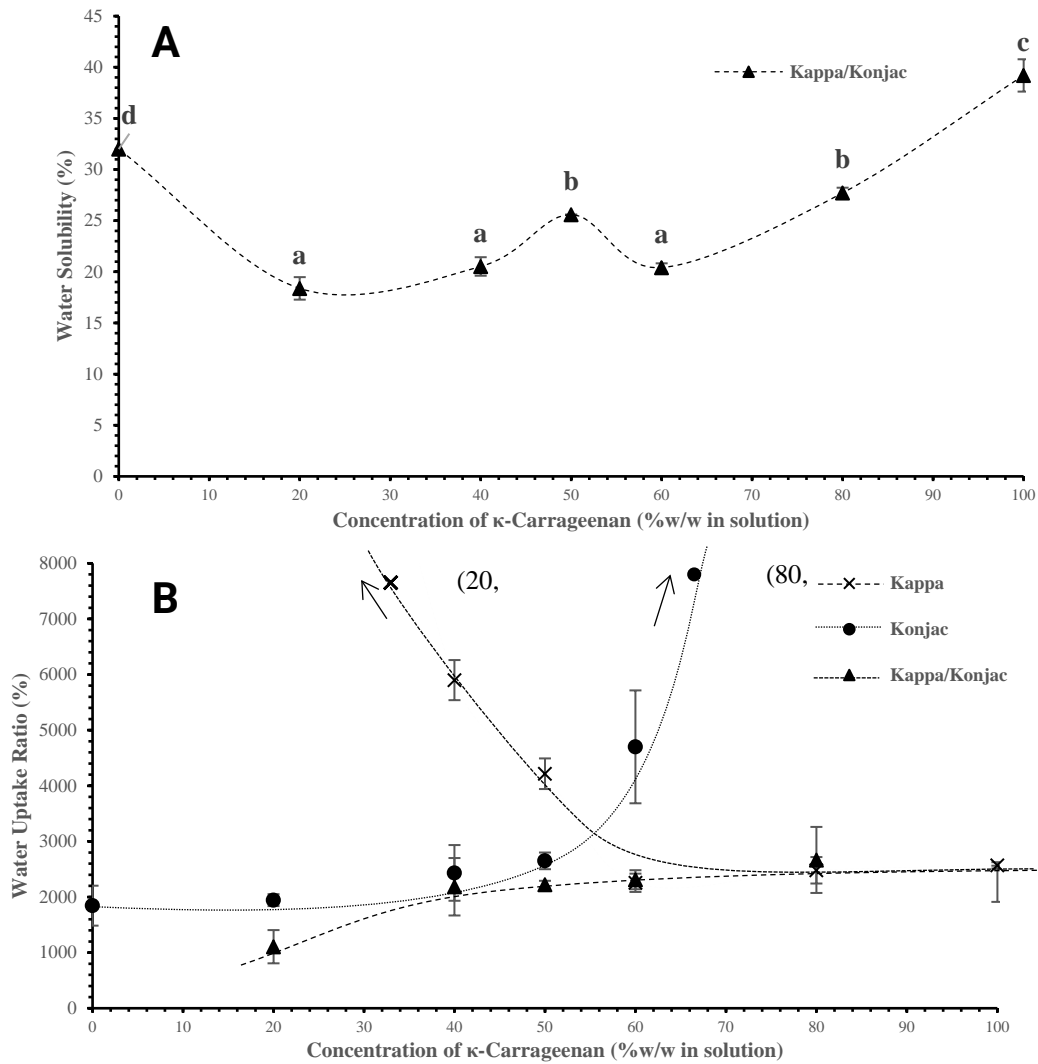


Figure 2. Water solubility (A) and Water uptake ratio (B) of different concentrations of κ -carrageenan (20% to 100%). Error bars show standard deviation.

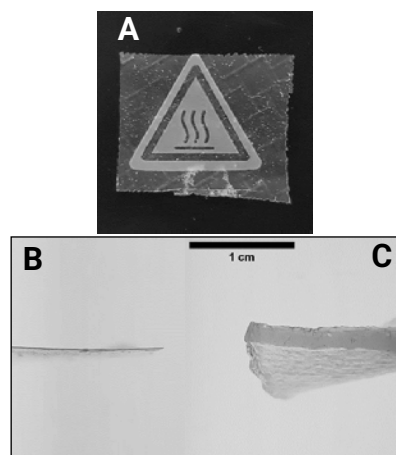


Figure 3. Different representations of the K4 film showing its transparency (A) when putted on a pictogram. (B) and (C) show the different thicknesses of the K4 film before and after being immersed in water, respectively.

Table 2. Water vapor permeability (WVP) of composite films of Konjac Glucomannan and κ -carrageenan at different concentrations and with PVC film. a–d Values with different letters within the same column are significantly different ($p < 0.05$). Data reported are means \pm standard deviation.

Film samples	WVP $\times 10^{-7}$ (g.Pa ⁻¹ .h ⁻¹ .m ⁻¹)
K6	4,39 \pm 0,34 ^{bc}
K5	5,63 \pm 0,38 ^{cd}
K4	6,66 \pm 0,42 ^d
κ -CG6	2,53 \pm 0,25 ^b
κ -CG5	2,70 \pm 0,48 ^{ab}
κ -CG4	2,69 \pm 0,26 ^{ab}
PVC film	1,01 \pm 0,09 ^a

3.5 Tensile strength and elongation at break

The mechanical properties of composite and monolayered films are shown in Figure 4. Tensile strength values were higher in composite films (35.11 – 56.85 MPa) than in monolayered films, as the films were too fragile to be tested. The addition of Glucomannan Konjac to the films tended to increase the tensile strength; however, the elongation at break of the samples decreased (0.98 – 5.25 %). These results seemed to be in accordance with the solubility. With less water available to play the role of a plasticizer, the film samples were less flexible, thus resulting in higher tensile strength and lower elongation at break. The network is, therefore, more solid but more brittle. Chen & al. [21] showed that if the composite film is humidified, acting as a gel, the network is more elastic and more flexible.

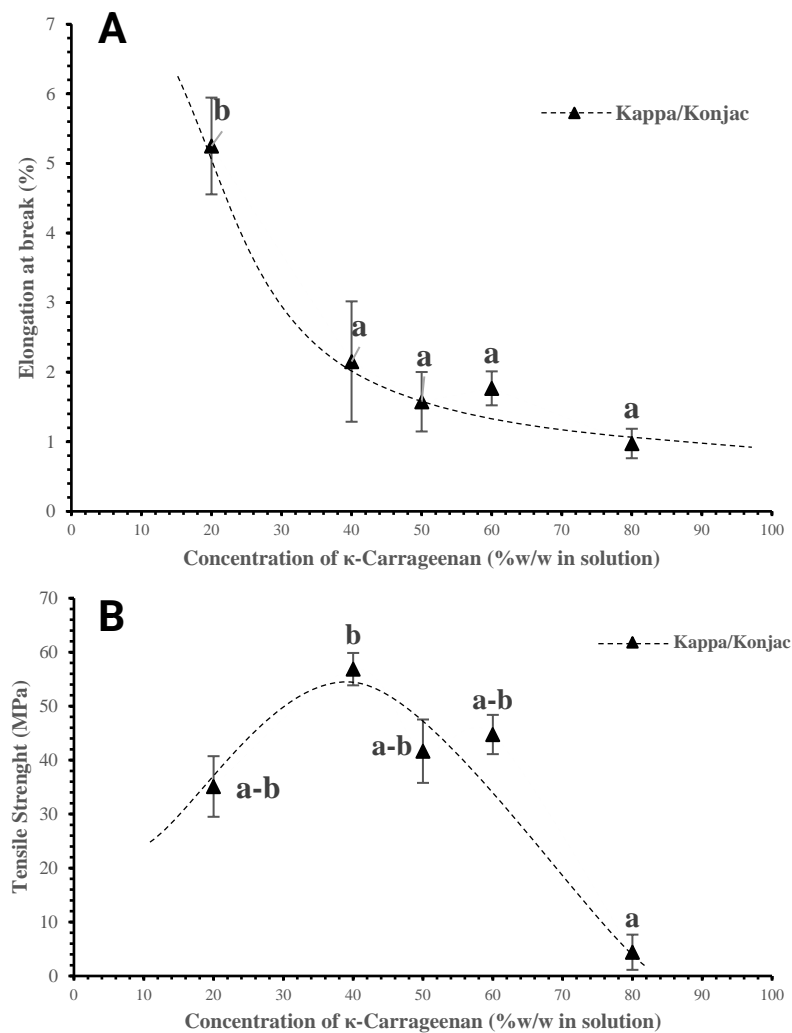


Figure 4. Elongation at break (A) and Tensile strength (B) of different concentrations of κ -carrageenan (20% to 80%). a–b Values with different letters significantly differ ($p < 0.05$). Error bars show standard deviation

3.6 FT-IR Analysis

FT-IR spectroscopy was employed to qualitatively analyze the chemical structure of the composite films, with the spectra of KGM, κ -CG, and K2 to K8 films presented in Fig. 5. The characteristic peaks of the KGM film at 3360 and 1034 cm^{-1} corresponded to the O-H stretching and C-O-C stretching vibrations, consistent with findings by Chen et al., 2019. Other peaks found at 1743, 1242, 1145 and 1060 cm^{-1} can be attributed respectively to C=O stretching, S-O sulfate stretching, C-O-H stretching and C-O-SO₃ stretching [28]. While new adsorption peaks were not observed in the spectra of the KGM: κ -CG film, a broadening of the O-H stretching peak was noted, indicating the formation of intermolecular hydrogen bonds between KGM and κ -CG [29], a variation of the intensity and forms of the sulfate related bands can be associated with their properties to make ionic interactions inside the composite [30]. The leading absorption bands of polysaccharides exhibited similarity, with peaks at 2924 cm^{-1} .

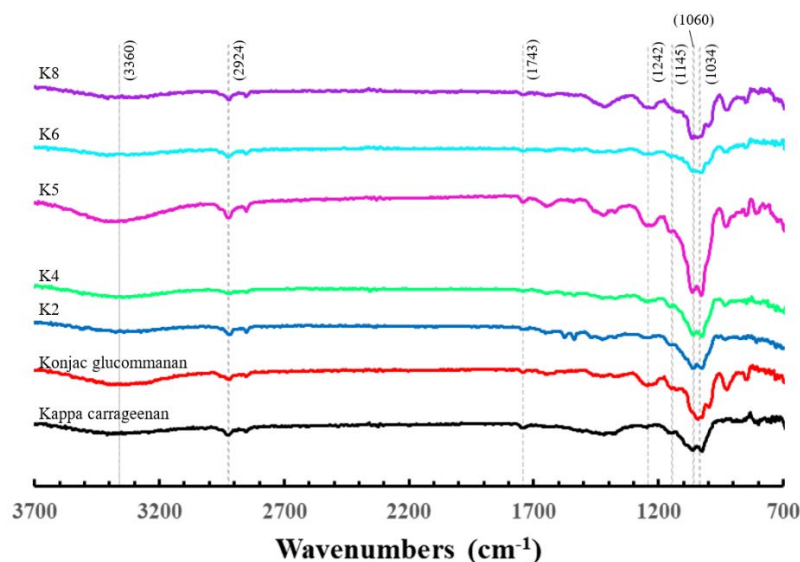


Figure 5. FT-IR spectra of the κ -CG /KGM blended films.

3.7 XRD Analysis

The X-ray diffraction of the blended films is depicted in Fig. 6. a. as well as KGM and κ -CG powders (Fig. 6. b and c.). High-ratio KGM films exhibited a characteristic noncrystalline broad peak around $2\theta = 20^\circ$, consistent with prior reports on the amorphous nature of pure KGM films [31] and as shown in Fig.6.c for pure KGM powder. In the κ -CG powder distinctive sharp peaks that cannot be associated with a specific structure. Another set of sharp peaks has been demonstrated by Qiao et al., 2023 but no concordance can be shown. The peaks can be associated with metal elements present during algae extraction, favoring a crystalline structure [32]. As we shift towards films with a higher concentration of κ -CG, XRD scans revealed a smaller peak at $2\theta = 15^\circ$. A small semi-crystallized peak showed around $2\theta = 30^\circ$ for K2 concentration. The more KGM is added, the broader the peaks, showing an amorphous structure in our films. The distinctive peaks observed in the raw material of highly crystallized pure κ -CG tend to diminish upon film formation, underscoring the transformative nature of the blending process. When associated with the functional groups of KG, the resulting structure remains amorphous, showing a potential interaction between the two polymers. Regardless of the composition, once films are formed, they exhibit an amorphous structure. While we observe nuanced variations in intensity between the peaks around 15 and 22°, it's essential to highlight that such variations do not allow us to make conclusive assertions regarding any substantial changes in crystallinity based on the KG/ κ -CG ratio.

4. Conclusion

Composite films made of Konjac Glucomannan and κ -carrageenan were successfully incorporated at different ratios (20:80 – 80:20 KGM κ -CG). The combination of konjac and carrageenan increased the thickness of the film until a specific ratio (50:50). The solubility of the composite films decreased, which could be linked to the interactions between the biopolymers' chains inside the network. These interactions improved the hydrophobic properties of these films. However, this property made the films stiffer, declining the mechanical properties, especially the elasticity with lower elongation at break. When a higher concentration of KGM was incorporated, WVPR decreased significantly ($p < 0.05$). With these results, the composite films could not be used as packaging

films because of their elasticity. The next approach is to incorporate by-product to make 3D object based on this film as a matrix.

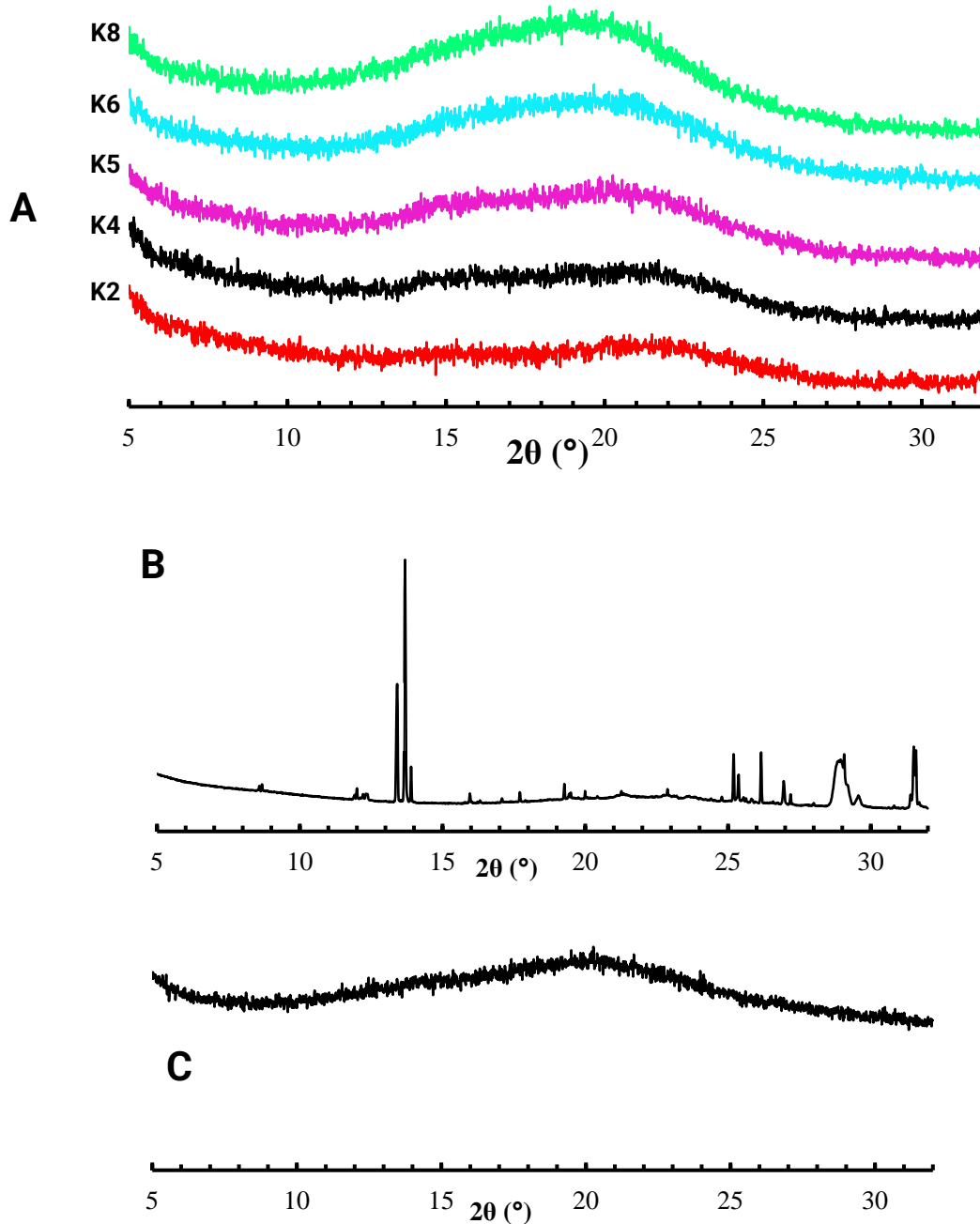


Figure 6. XRD curves of the κ -CG /KGM blended films (A), κ -carrageenan powder (B) and konjac glucomannan powder (C).

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