



KAPPA CARRAGEENAN POLYMERIC FILMS: A COMPREHENSIVE STUDY ON SYNTHESIS AND CHARACTERIZATION

AHUJA S, PATEL P, RAULJI H, AND PAWAR Y

Department of Chemistry, Parul Institute of Applied Science, Parul University, Waghodiya, Gujarat, India

*Corresponding Author: Dr. Sonam Ahuja: E Mail: sonam.ahuja82106@paruluniversity.ac.in

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ABSTRACT

Kappa carrageenan, a biopolymer derived from red seaweeds, a biopolymer that has drawn a lot of interest because of its special qualities and broad range of uses. The goal of this work is to provide a better knowledge of the structural and thermal properties of kappa carrageenan polymeric films by the synthesis and thorough characterization of these films. These properties were investigated using a variety of analytical techniques, such as X-ray diffraction (XRD), thermogravimetric analysis (TGA), scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and an examination of the behavior of these films during gelatin expansion. The FTIR study provided important details regarding the functional groups and chemical makeup of the kappa carrageenan films. SEM pictures gave information on the films' microstructure and surface morphology as well as visible proof of their structural characteristics. Thermal stability, degradation temperatures, and the existence of any additives or contaminants in the films could all be evaluated thanks to TGA data. The films' degree of crystallinity and crystalline character were clarified by XRD examination. Additionally, the study investigated the relationship between gelatin, a common biopolymer, and kappa carrageenan films. In order to comprehend the suitability and possible uses of these films in the food and pharmaceutical industries—two fields that frequently use mix materials—this interaction was evaluated. The findings of this study may have a big impact on how new packaging materials, medication delivery methods, and biodegradable items are developed. The present study's synthesis and characterisation of kappa carrageenan polymeric films offer a thorough comprehension of their characteristics, paving the way for a variety of uses in many sectors. These discoveries add to the expanding field of sustainable and biodegradable materials, which may provide answers to today's environmental problems.

Keywords: Kappa Carrageenan, FTIR, TGA, SEM, XRD

1.0 INTRODUCTION

The pressing global need for ecologically friendly, sustainable materials has fueled the development of biodegradable polymers [1], particularly for uses in packaging, food preservation, and biomedical disciplines. Because of its biodegradability, biocompatibility, and non-toxicity, kappa carrageenan, a naturally occurring polysaccharide produced from red seaweeds, has received substantial interest as a promising biopolymer [2]. As the globe struggles with the growing problems of plastic waste and environmental deterioration, the environmental consequences of conventional plastics, such as their non-biodegradability and contribution to plastic pollution, have created an urgent demand for alternative materials. With its sustainable sourcing from several seaweed species, kappa carrageenan stands out as a viable alternative, offering not just biodegradability but also a lower ecological footprint throughout its life cycle. These properties make it an excellent choice for the creation of ecologically friendly packaging materials as well as a variety of biological applications. kappa carrageenan has emerged as an eco-friendly alternative with distinct advantages. There has been a growing amount of material in recent years demonstrating the possibilities of carrageenan-based films. Researchers have highlighted their film-forming ability, mechanical strength, and

exceptional barrier characteristics against gases and moisture [3], the ready availability of kappa carrageenan from various seaweed sources ensures a sustainable supply chain for the manufacturing of biodegradable products [4]. Despite the potential, widespread use of kappa carrageenan-based films has been hampered by limitations such as their susceptibility to environmental conditions such as humidity and temperature, which can have a substantial impact on their mechanical and barrier properties. To fully realize the potential of these biopolymeric films, it is critical to investigate their synthesis procedures and conduct extensive characterization. Here we have made a polymeric film of kappa carrageenan and to see its chemical analysis we have done gelatine expansion and to this work includes a thorough investigation into the synthesis process, with a focus on the effect of crucial parameters like as concentration, temperature, and the usage of cross-linking agents on the structural and functional properties of kappa carrageenan films. Characterization techniques such as scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and thermogravimetric (TGA) analysis are used to examine the polymeric films' morphology, chemical structure, thermal stability [5]. This study aims to contribute to the evolution of sustainable packaging materials and

biomedical applications by deepening our understanding of the structure-property relationships in kappa carrageenan-based films and facilitating their integration into various industrial sectors. The synthesis and characterisation of kappa carrageenan polymeric films represents an important step toward a more sustainable future as we stand at the crossroads of environmental responsibility and scientific progress.

2.1 MATERIAL AND METHOD

2.2 Material:

Sisco Research laboratories pvt.ltd, Taloja, Maharashtra (India) provided Kappa-carrageenan, Distilled water.

2.3 Method:

2.3.1 Preparation of kappa carrageenan polymeric film:

To prepare a 1% weight/volume solution of kappa carrageenan in powder form, 0.5 grams of kappa carrageenan should be measured and added to 50 milliliters of distilled water. This mixture should be placed on a magnetic stirrer and stirred for 10 to 15 minutes until the kappa carrageenan is completely dissolved. Once dissolved, the resulting solution can be transferred to a glass petri dish and allowed to dry at room temperature for a period of 24 to 48 hours.

3.1 Chemical Analysis:

3.2 Gelatine Expansion Study:

The expansion of the wound dressing film on the wound surface was investigated using the change in diameter of a circular film sample

in a 10% gelatine solution. To put it briefly, 10 g of gelatine powder were dissolved in 100 ml of heated distilled water, with constant stirring until a clear solution was obtained. The gelatine solution was then added to all ratio films with known diameters, which were placed in a petri dish. The diameter change was then continually monitored until the sample's diameter stabilized.

The expansion ratio (ER) was determined using the formula $ER = D_t/D_o$, where D_t is the diameter at time t and D_o is the original diameter [6].

4.1 Characterization:

4.2 FTIR analysis:

FTIR analysis can offer a thorough knowledge of the chemical and structural characteristics of biodegradable polymeric films when combined with other analytical methods. Fourier transform infrared spectroscopy, or FTIR, is a technique for determining a kappa carrageenan polymeric film's composition. The spectra ranged from 4000 to 400 cm^{-1} after being dried in an oven for 24 hours with 1% of each polymer present. The FTIR analysis performed at Parul University's Central Research for Development [7].

4.3 SEM analysis:

An electron microscope known as a scanning electron microscope (SEM) scans a sample's surface by taking images of the material with a focussed electron beam. The surface topography and chemical makeup of the

sample are revealed by the many signals produced by the electrons' interactions with the atoms in the sample. SEM analysis was done to assess the surface morphology and figure out how the composite films spread. Granular form analysis was performed on the ground-up composite film. The working distance (WD) of the electron beam was maintained at 6.6 mm, and the accelerating voltage (HV) of the electron beam was set to 5.00 kV [8].

4.4 Thermal gravimetric analysis:

When controlled heating is provided, TGA analysis calculates the mass of the experiment as a function of temperature or time. TGA is a material analysis technique. When the material's constituent elements progressively volatilize over time, temperature and weight loss are observed. Because TGA testing can measure weight loss at very high temperatures, it is a useful technique for evaluating polymers. Although certain polymers can withstand temperatures of 301°C in air and 505°C in inert gasses without deteriorating, most polymers melt at temperatures of around 200°C before

breaking down. TGA is an additional tool for studying these polymers. Thermal gravimetric analysis performed at Department of chemistry at Sardar Patel University, Anand [9].

4.5 XRD (X-ray Diffraction) Analysis:

In materials science, chemistry, geology, and many other scientific fields, X-ray diffraction (XRD) is a potent analytical method that is frequently used to examine the atomic and molecular structure of a crystalline substance. This non-destructive technique, which provides important details regarding crystal structure, orientation, grain size, and phase composition, is based on the principles of X-ray scattering by crystalline materials [10].

5.1 RESULT AND DISCUSSION:

5.2 Gelatine Expansion study:

Here in **Table 1, Figure 1**, expansion studies are carried out to evaluate the diameter growth of polymer films. The subsequent phrase was employed to ascertain the ER, or expansion ratio:

$ER = D_t/D_o$, where D_t is the diameter at time t and d_o is the starting diameter

Table 1: Gelatine Expansion Study

Time	Expansion of Pectin film
0 min	1
20 min	1.5
40 min	1.7
60 min	1.9
80 min	2.4

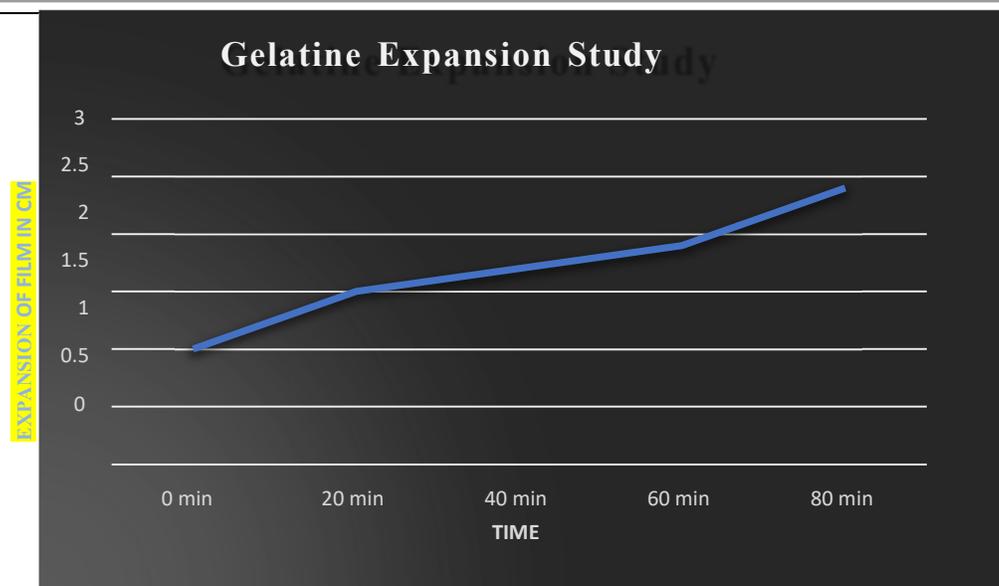


Figure 1: Graph of Gelatine Expansion

5.3 FTIR Analysis:

Here in **Figure 2**, In the FTIR spectrum of kappa carrageenan, the wide band at 3356.93 cm^{-1} corresponds to the stretching vibrations of hydroxyl (OH) groups and hydrogen-bonded water molecules. It confirms the presence of (OH) group within the film, this wide peak attests to the existence of hydroxyl groups that are free and those that form hydrogen bonds with water molecules. The peak at 1639.04 cm^{-1} in the FTIR confirms the carbonyl (C=O) stretching vibration of ester groups within the carrageenan structure. This peak is typical of the uronic acid residues found in carrageenans, and it is frequently visible in their FTIR spectra. The peak at 1230.71 cm^{-1} in the FTIR (Fourier-transform infrared spectroscopy) spectrum of kappa carrageenan polymeric film is associated with the stretching vibration of sulfate (S=O) groups

within the carrageenan structure. This peak represents the presence of sulfate ester linkages in kappa carrageenan. The existence of ether linkages (C-O-C) within the carrageenan structure was confirmed with the peak at 1040.32 cm^{-1} in the FTIR. The stretching vibrations of the C-O-C bonds are represented by this peak. Like many other polysaccharides, kappa carrageenan contains these ether linkages as part of its sugar unit structure.

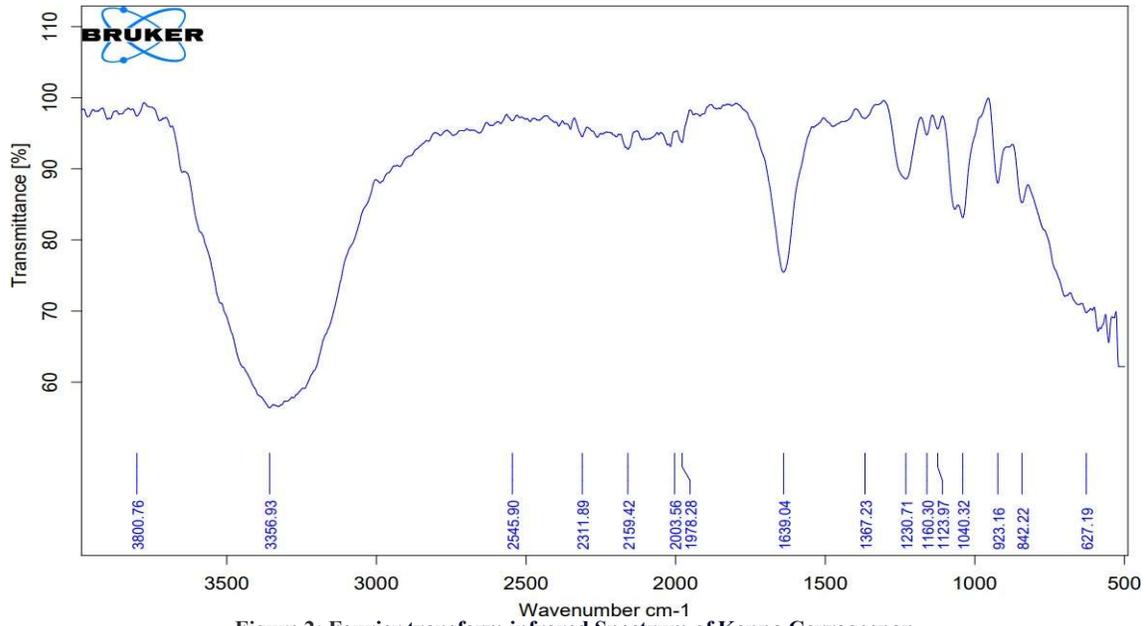


Figure 2: Fourier transform infrared Spectrum of Kappa Carrageenan

5.4 Thermal Gravimetry Analysis:

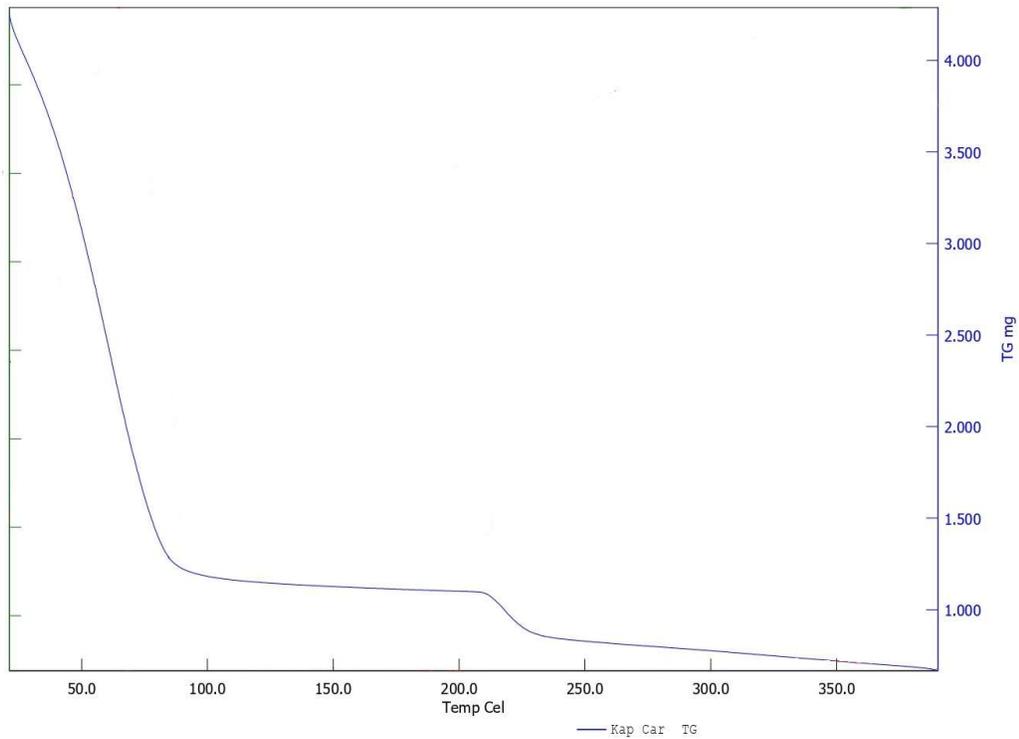


Figure 3: Thermal Gravimetry Analysis Graph

Here in the **Figure 3**, the heat stability and decomposition behavior of the kappa carrageenan polymeric film are revealed by the TGA analysis. The graph shows how the weight of the sample changes with increasing temperature, which is important for understanding its thermal properties and prospective applications, the temperature program for Kappa Carrageenan, a sample weighing

4.307 mg, was conducted at a rate of 10°C/min, using nitrogen gas at a flow rate of 150 mL/min. The TGA study was carried out at temperatures ranging from 30°C to 400°C. The initial temperature was 30.0°C, which increased at a constant rate of 10°C per minute, and the analysis concluded at 400.0°C. The TGA graph depicts the weight loss of the heated kappa carrageenan polymeric film. At 30°C, the sample's starting weight was 4.307 milligrams. The sample started to lose weight as the temperature rose. The graph illustrates the weight loss and the mass loss or rate of decomposition at various temperatures. The greatest loss of weight happened at about 300°C. At 400°C, the final weight was roughly 1.000 mg.

5.4 SEM analysis:

Here in **Figure 4**, an examination of a polymeric film derived from kappa carrageenan using SEM (Scanning Electron Microscopy) can provide important details about the film's surface properties and shape. The surface topography and texture of the

polymeric film may be seen using SEM. It can display the film's surface characteristics, such as smoothness, roughness, porosity, or texture, which is crucial for determining whether or not it is appropriate for a certain use, such as coatings or packaging. The electron high tension, or voltage applied to the electron source in the SEM, is represented by the "EHT" value of 10.00 kV. This voltage affects the energy of the electrons utilized in imaging, which can have an effect on the depth and clarity of the pictures that are produced. Signal A" is linked to certain configurations employed in the SEM examination. The magnification level was set at 5.00 KX (kiloX), indicated by the notation "InLens Mag". The distance at which the electron beam concentrated for imaging in this instance was kept at 4.0 mm, or the working distance. This SEM analysis shown in **Figure 4**.

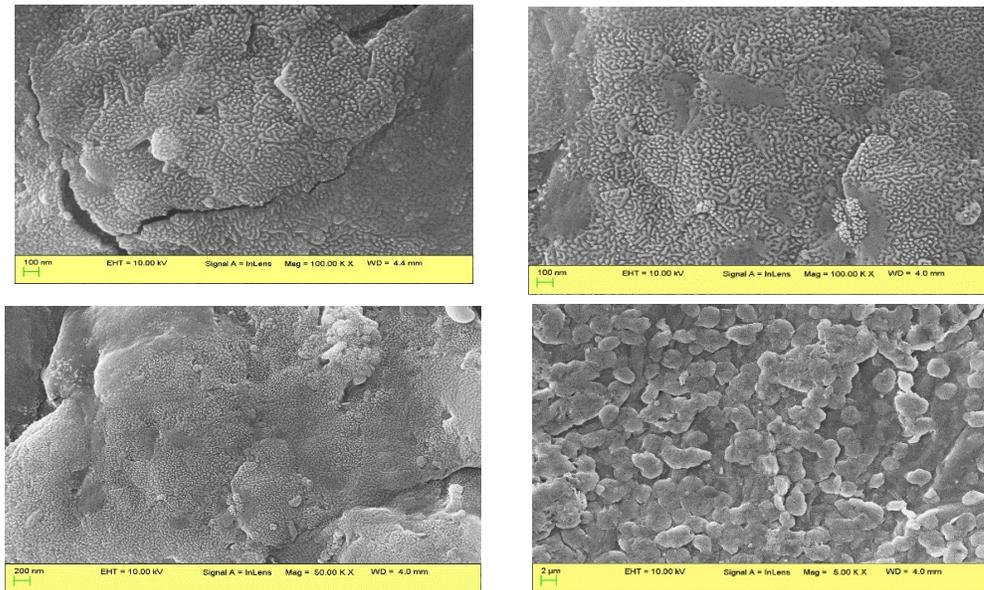


Figure 4: Scanning Electron Microscopy Analysis

5.5 X-ray Diffraction Analysis:

Here in the **Figure 5**, the intensity value corresponds to a conspicuous peak located at a diffraction angle of 64.76234 degrees. The intensity scale has a range of 0 to 70 arbitrary units (a.u.), with 70 a.u. being the highest intensity point. Several peaks show organized atomic configurations within the crystal lattice, and the overall pattern indicates the crystalline structure of the material. The intensity values give information regarding the

abundance of various crystallographic planes by indicating the amount of X-rays diffracted at each particular angle. From 0 to 90 degrees, the diffraction angles span the entire range of crystal orientations. The intensity values measure the degree of crystallinity, whereas the presence and location of peaks at particular 2θ values provide information on the crystal structure. The areas with reduced diffraction or amorphous phases are indicated by the baseline between peaks.

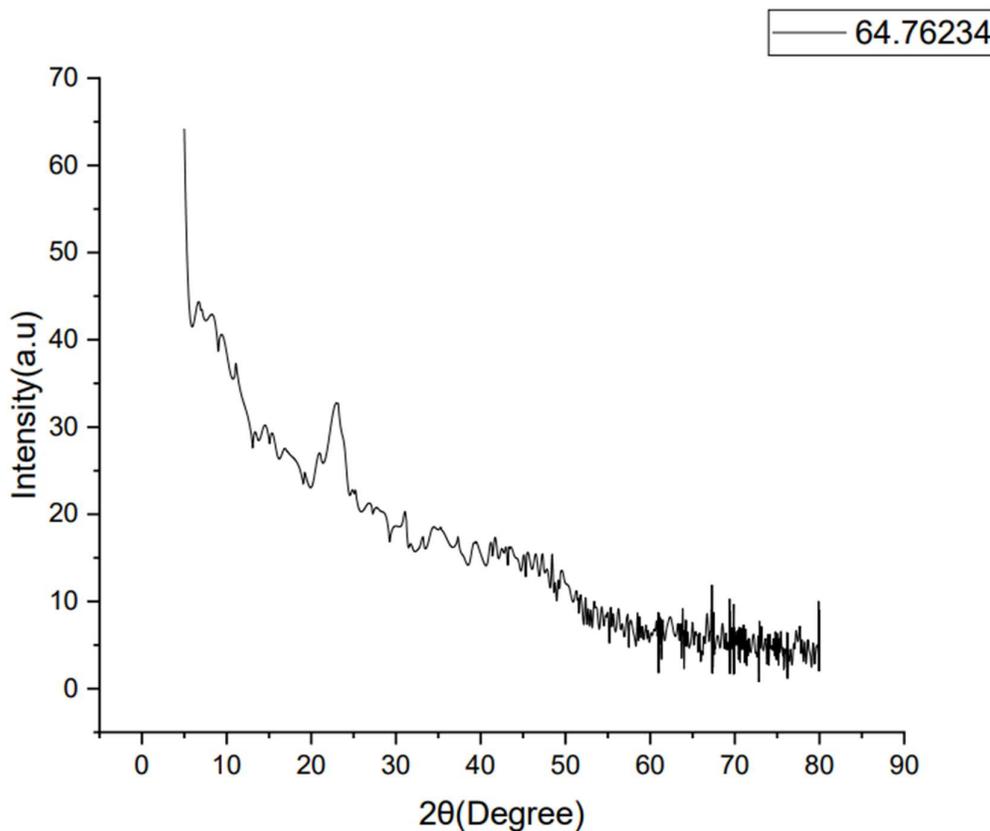


Figure 5: XRD GRAPH

CONCLUSION:

To sum up, our study's synthesis and in-depth analysis of kappa carrageenan polymeric films provide important new understandings of their thermal and structural characteristics. The study, which used methods like FTIR, SEM, TGA, and XRD, has given us a thorough understanding of these films. The study explores how kappa carrageenan might be used as a sustainable substitute in a number of industries, including packaging and biological uses. The dynamic behavior of the kappa carrageenan film was shown by the gelatine expansion investigation, suggesting that it is suitable for use in wound dressing applications. The film's responsiveness and

possible adaptation in a variety of settings were demonstrated by the expansion ratio's gradual increase. Additionally, the films' chemical composition was fully disclosed by the FTIR analysis, and their surface morphology and microstructure were shown by the SEM. Understanding thermal stability, degradation temperatures, and the existence of any additives or impurities was made possible by TGA data. The results highlight how crucial it is to optimize synthesis parameters, such as temperature and concentration, in order to improve the structural and functional characteristics of kappa carrageenan films. This research is in line with the global search for environmentally friendly alternatives amid

worries about plastic waste and environmental degradation. It also makes a significant contribution to the development of sustainable packaging materials and biomedical applications, despite environmental challenges.

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Conflict of Interest

No conflicts of interest exist, according to the authors, with the publishing of this work

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