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DEVELOPMENT AND CHARACTERIZATION OF SOY PROTEIN ISOLATE–MONTMORILLONITE BIODEGRADABLE FILMS

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ABSTRACT

Biodegradable polymer-clay nanocomposites are the subject of recent surge in study due to the increasing need for environmentally acceptable and sustainable packaging materials. Research in this area focuses on biodegradable films made of soy protein isolate (SPI) reinforced with montmorillonite (MMT) nanoclay at concentrations ranging from 0% to 5%. We methodically investigated the films' structural, mechanical, and barrier characteristics after they were created using a solution-casting process. As the clay content increased, the film thickness also rose somewhat, from 0.081 mm (SPI-0) to 0.093 mm (SPI-5). A greater MMT content was shown to significantly increase tensile strength (18.4 to 29.0 MPa) and Young's modulus (320 to 720 MPa) in mechanical study, whereas a comparable drop in elongation at break (56.3% to 28.5%), suggesting enhanced rigidity but lower flexibility, was observed. A thorough examination of the barrier revealed a significant decrease in water vapor permeability (6.25 to 3.60 g•mm/m²•day•kPa), moisture content, and solubility, validating the beneficial impact of clay inclusion on moisture resistance.

Keywords: Montmorillonite, Biodegradable Films, Nanocomposite, Mechanical, Plasticizer.

I. INTRODUCTION

The hunt for environmentally friendly alternatives to traditional petroleum-based plastics has heated up in response to the worldwide worsening of pollution from these products. Biodegradable polymers have attracted a lot of interest from scientists and businesses in the past 10 years because to their potential usage in many different fields, including agriculture, throwaway items, food packaging, and biomedicine. Because of their film-forming capacity, biodegradability, renewability, and physicochemical flexibility, proteins generated from plants have stood out as one of the most promising biopolymers thus far. With a protein content of over 90%, Soy Protein Isolate (SPI) is a natural polymer that shows great promise for creating environmentally friendly films. Biodegradable packaging materials may be made using SPI due to its vast availability, low cost, nutritional quality, and functional qualities including emulsification, water-binding, and structural flexibility. Unfortunately, the practical use of pure SPI films is limited because to their brittleness, lack of mechanical strength, and low water resistance.

Montmorillonite is a smectite clay family member and a naturally occurring layered silicate mineral with a remarkable surface area, high aspect ratio, and distinctive nanostructure. The production of nanocomposites with vastly improved mechanical, thermal, and barrier characteristics is made possible by its plate-like shape, which allows for easy intercalation with polymer matrices. One of the main reasons sodium-based MMT is highly regarded is its capacity to improve stress transfer, limit the mobility of polymer chains, and decrease film permeability through exfoliation or intercalation inside biopolymer networks. Hydrogen bonding, electrostatic attraction, and trapping of polymer chains are the mechanisms by which MMT nanosheets interact with an SPI matrix, enhancing the composite film's structural reinforcement. A novel family of eco-friendly materials is born from the complementary properties of protein-based polymers and inorganic nanoclays; these materials are both biodegradable and highly functional.

There is a strong global priority to produce sustainable packaging materials that minimize environmental effect, reduce plastic waste creation, and promote circular economic models. The development of SPI/MMT nanocomposite films is in great alignment with this goal. Biopolymers made from agricultural resources, such soy proteins, have several benefits over plastics made from petroleum, which end up in land and marine environments for centuries. One of these advantages is that they are renewable and biodegradable. Because of their natural oxygen barrier qualities, which aid in preventing oxidative degradation of packed goods, films based on SPI have also demonstrated great potential in food packaging applications. The integration of nanoclays, such as MMT, can greatly alleviate the problems caused by their hydrophilic character, which results in high water vapor permeability and low resistance to humidity. Multiple studies have shown that even small amounts of MMT may significantly enhance the barrier qualities of SPI films. This is because it creates a convoluted diffusion route, which slows down the transfer of gases and water vapor.

Plasticizers, such as glycerol, play an important role in determining how well SPI/MMT films work. By decreasing intermolecular pressures and boosting mobility of polymer chains, plasticizers improve the processability, extensibility, and flexibility of films based on proteins. The lack of proper plasticization renders SPI films almost useless due to their brittleness. On the other hand, there is a cost to using glycerol; too much plasticizer lowers mechanical strength and water resistance. Producing films that fulfill particular application needs thus requires an ideal combination of polymer, plasticizer, and reinforcing nanofiller. The film's ultimate morphology, crystalline structure, mechanical behavior, and barrier properties are all dictated by how these components interact with one another. As more and more sectors look for biodegradable alternatives with competitive performance qualities, understanding and improving these interactions has become a significant focus of present research.

Common methods for making SPI/MMT films include solution casting, which entails dissolving SPI in plasticizer, heating it to cause partial denaturation, mixing it with dispersed MMT clay, and then casting the mixture into thin films. As the clay layers are distributed and the protein matrix undergoes conformational changes, the composite's microstructural and functional characteristics are profoundly affected. To better understand the molecular interactions, intercalation behavior, and morphology of the nanocomposite structure, it is common practice to use advanced characterization methods. These methods include X-ray diffraction (XRD), thermogravimetric analysis (TGA), scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FTIR). Researchers may use these analytical insights to learn how much MMT improves mechanical strength, thermal stability, and barrier characteristics, and what the possible downsides, such less film transparency or more stiffness, of increasing the clay content are.

Rising environmental regulations, consumer desire for sustainable packaging, and technical breakthroughs in nanocomposite production have all contributed to a dramatic expansion of the possible uses of SPI/MMT biodegradable films in recent years. The versatility of films as edible coatings, biodegradable wrapping, or protective layers with gas barrier qualities means that food packaging is still a hot topic. Furthermore, nanocomposites based on SPI have been studied for use in wound dressings, controlled-release systems, and tissue engineering scaffolds because of its non-toxicity and biocompatibility. Because of their biodegradability and environmental compatibility, SPI/MMT films have several potential applications in agriculture, including mulch films and seed coatings. Sustainable products are becoming more important in global markets, and SPI/MMT nanocomposites provide an attractive alternative to conventional plastics without sacrificing performance.

II. REVIEW OF LITERATURE

Sharma, Shreya et al., (2023) For several reasons, including its availability, low cost, eco-friendliness, film-forming potential, and processability, soy protein isolate has been the center of interest in the last few decades. Innovative bionanocomposite films based on soy protein isolate (SPI) were created in this work by utilizing a straightforward solution casting method to include Mg-Allayered double hydroxide (LDH) at varying loadings (0%, 2%, 5%, and 9% w/w). Using a co-precipitation technique, the Mg-Al LDH was produced in a 2:1 molar ratio. In the SPI matrix of bionanocomposite films, Mg-Al LDH sheets were found to be intercalated or exfoliated, according to FTIR, XRD, field-emission scanning electron microscopy, and thermogravimetric analysis (TGA). Concentrations ranging from 0% to 9% (w/w) of Mg-Al LDH caused noticeable agglomeration. A high level of thermal stability was observed in the bionanocomposite films. They looked at its biodegradability and mechanical properties as well. The films that were made with 0%, 2%, 5%, and 9% w/w LDH loadings had tensile strength values of 2.12 ± 0.25, 1.60 ± 0.15, 1.64 ± 0.08, and 1.58 ± 0.06 KNm/g, respectively. These values were recorded using standard statistical methods. An substantial improvement in tensile strength was seen in the SPI-Mg/Al LDH 5% film, which also decomposed well in non-sterile soil.

Roufegarinejad, Leila. (2022) An active packaging method including soy protein isolate (SPI) nanocomposite film with copper oxide (CuO) and titanium dioxide (TiO_2) nanoparticles (NPs) was the focus of this scholarly investigation. Film morphology analysis revealed that NP integration resulted in a compact and dense structure. Using Fourier transforms infrared structural conformations, the interaction generation between the SPI matrix and NPs was authorized. Adding NPs to SPI-based film increased its mechanical characteristics. Film samples containing 1% CuO and TiO2 NPs had the lowest moisture content, water solubility, and water vapor permeability. The antioxidant activity in the films based on SPI was further enhanced by the addition of both NPs. Incorporating NPs resulted in a substantial decrease ($p < 0.05$) of the L* color parameter while increasing the a* and b* color parameters. In addition, the opacity of the SPI-based films was enhanced by adding CuO and TiO2 NPs. Finally, the physicochemical characteristics of SPI-based films were improved by adding CuO and TiO2 NPs, and the resulting nanocomposite films have potential as an active packaging method for various foods.

Qin, Zhiyong et al., (2019) Because of its biodegradability, excellent film-forming properties, and ease of processing, soy protein isolate (SPI) has garnered a lot of interest from the packaging technology community. Nevertheless, the limited practical use of films based on SPI is typically due to their poor mechanical qualities and extreme susceptibility to moisture. Here, a biobased nanocomposite film was created by cross-linking an SPI matrix with cellulose nanofibers (CNF) and nano-silica (NS) particles, which work together as a synergistic reinforcement. To get the most out of the epoxy-dominated cross-linking process, which improves the interfacial contact between SPI and CNF/NS, we started by functionalizing the CNF with NS using a silane agent (KH560). Advanced imaging techniques such as Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), and thermogravimetric analysis (TGA) were used to conduct a thorough investigation of the morphology, thermal stability, and chemical composition of the resulting nanocomposite films. These findings provided evidence of effective surface modification and demonstrated that the surface-tailored CNF/NS nanohybrid efficiently adheres to the SPI matrix via hydrogen-bonding and covalent interactions. Incorporating CNF/NS into SPI produced nanocomposite films that outperformed the pure SPI film in terms of tensile strength (6.65 MPa), an improvement of 90.54%. The water contact angle (91.75°) was greater and the water vapor penetration was much lower in the resultant composites compared to the unaltered film.

Jensen, A. et al., (2015) This research used a hot surface casting method to create composite films consisting of cellulose and soy protein isolate (SPI). A combination of acid-alkaline hydrolysis and high-pressure homogenization was used to remove the cellulose microfibrils from soybean pods and stems for the films. In order to compare, composite films made with TiO2-SPI filler were also created. Various relative humidity (RH) conditions were used to assess the mechanical and barrier characteristics, including water vapor and oxygen permeability, of these files. Raising the RH had a negative effect on tensile strength (TS) and Young's modulus (YM), whereas raising EAB had the opposite effect. The 5 g:95 g fiber:SPI film showed a notable improvement in TS and YM, but a loss in EAB, as compared to the clean SPI, among the studied composites. In contrast, the control film and the TiO2 composites had identical TS, YM, and EAB values. The barrier characteristics were similar in all samples and were worse as the relative humidity got higher. The samples were examined with atomic force microscopy, scanning electron microscopy, and Fourier transform infrared spectroscopy.

Kumar, Parikshit et al., (2010) There has been a resurgence of interest in bio-nanocomposites, which are biopolymer matrices reinforced with nanoparticles such layered silicates, as a potential replacement for plastic packaging due to its indegradability and lack of recycleability. I used melt extrusion to create bio-nanocomposite films using soy protein isolate (SPI) and montmorillonite (MMT). We looked examined how the structure and characteristics of SPI-MMT bio-nanocomposite films were affected by various extrusion processing factors, including the pH of the film forming solution, the amount of MMT, and the screw speed and barrel temperature distribution. Film structure was studied using X-ray diffraction (XRD), transmission electron microscopy (TEM), and scanning electron microscopy (SEM). Thermal gravimetric analysis (TGA), tensile testing, and water vapor barrier measuring were used to assess the films' properties. For soy protein matrices, MMT arrangements varied from exfoliated at 5% MMT concentration to intercalated at 15% MMT content or higher. When MMT was added to the films, their mechanical and dynamic mechanical parameters, including tensile strength, % elongation at break, storage modulus, glass transition temperature, thermal stability, and water vapor permeability, were improved. The study's findings demonstrate that bio-nanocomposite technology can enhance the characteristics of SPI-based biopolymer films.

III. MATERIALS AND METHODS

Materials

A commercial provider provided the ingredients utilized to make the biodegradable films, which comprised food-grade soy protein isolate (SPI) with around 90% protein. The reinforcing nanofiller choose was sodium-based montmorillonite (MMT) clay, which has a particle size less than 2 µm, because of its large surface area and compatibility with biopolymer matrices. To enhance the film's flexibility and handling qualities, analytical-grade glycerol was used as the plasticizer. To guarantee purity and prevent interference during film creation and characterization, all solutions and dispersions were made using distilled water.

Film Preparation

After 30 minutes of magnetic stirring at 60 °C, SPI was dispersed in distilled water to make a 5% (w/v) solution. The plasticizer glycerol was added at a rate of 30% (w/w of SPI). Following a 30-minute sonication of MMT in water, it was mixed with SPI solution to produce MMT concentrations of 0% (control), 1%, 3%, and 5% (w/w, according to SPI mass). Following 48 hours of drying at 25 °C and 50% RH, the solutions were carefully transferred on leveled glass plates. Before testing, the dried films were subjected to 48 hours of conditioning at 25 °C and 50% RH after being peeled.

Film Thickness

The average film thickness was determined by measuring it five times at random using a digital micrometer.

Mechanical Properties

Measurements of tensile strength, elongation at break, and young's modulus were taken using an Instron texture tester in accordance with ASTM D882. At a crosshead speed of 50 mm/min, samples measuring 10 mm × 100 mm were subjected to testing. Five separate formulations were conducted.

Water Vapor Permeability (WVP)

The ASTM E96 gravimetric technique was used for the measurement of WVP. Desiccators set at 75% relative humidity were used to seal films over cups containing silica gel. We measured weight growth over time and estimated WVP (g•mm/m²•day•kPa) while accounting for film thickness.

Moisture Content (MC) and Solubility

MC was calculated by drying film samples to a constant weight at 105 °C. Tests for water solubility include soaking film samples in distilled water at 25 °C for 24 hours, removing the insoluble portion, and then determining the percentage of solubility.

Statistical Analysis

Five separate experiments were carried out for each type of study. The data is given as the average plus or minus the standard deviation. To identify formulation-specific differences, one-way ANOVA was employed, followed by Tukey's post-hoc test ($\alpha = 0.05$).

IV. RESULTS AND DISCUSSION

Film Thickness

Table 1: Film thickness

Formulation	MMT (%)	Thickness (mm, mean ± SD)
SPI-0	0	0.081 ± 0.004
SPI-1	1	0.084 ± 0.003
SPI-3	3	0.087 ± 0.005
SPI-5	5	0.093 ± 0.006

Film thickness is positively correlated with MMT concentration, as seen in Table 1. The SPI-0 control film, which has no metal matrix transitions, is 0.081 ± 0.004 mm thick. The thickness goes up to 0.084 ± 0.003 mm with 1% MMT (SPI-1), and it goes up even more to 0.087 ± 0.005 mm with 3% MMT (SPI-3). In SPI-5 with 5% MMT, the maximum thickness of 0.093 ± 0.006 mm is recorded.

Mechanical Properties

Table 2: Tensile properties of SPI/MMT films

Formulation	**Tensile Strength (MPa)**	**Elongation at Break (%)**	**Young's Modulus (MPa)**
SPI-0	18.4 ± 1.2	56.3 ± 4.8	320 ± 25
SPI-1	22.1 ± 1.5	48.7 ± 3.9	410 ± 30
SPI-3	27.8 ± 1.8	35.2 ± 3.1	640 ± 45
SPI-5	29.0 ± 2.0	28.5 ± 2.6	720 ± 60

The impact of montmorillonite addition on the mechanical characteristics of films based on SPI is shown in Table 2, where an increase in the clay content is associated with a noticeable improvement in stiffness and tensile strength. The tensile strength of the control film (SPI-0) was 18.4 ± 1.2 MPa. However, when MMT was added, the film's tensile strength rose gradually, reaching 22.1 ± 1.5 MPa at 1% loading (SPI-1) and 27.8 ± 1.8 MPa in the SPI-3 formulation. The SPI-5 material had the greatest tensile strength, measuring 29.0 ± 2.0 MPa. In SPI-5, Young's Modulus grew from 320 ± 25 MPa in SPI-0 to 720 ± 60 MPa, following a similar pattern. On the other hand, as the MMT concentration increased, the elongation at break decreased. In the control film, it was 56.3 ± 4.8%, but in SPI-1, it dropped to 48.7 ± 3.9%. In SPI-3, it fell even lower to 35.2 ± 3.1%, and in SPI-5, it fell to 28.5 ± 2.6%. A one-way ANOVA test verified that MMT had a highly significant impact on tensile strength, elongation, and modulus ($p < 0.001$). Higher clay loadings greatly mechanically strengthen the SPI matrix, as shown by Tukey's post-hoc test, which also showed that SPI-3 and SPI-5 were statistically different from SPI-0 and SPI-1.

Barrier Properties (WVP)

Table 3: Water vapor permeability and moisture-related properties

Formulation	**WVP ($g·mm/m^2·day·kPa$)**	**Moisture Content (%)**	**Solubility in Water (%)**
SPI-0	6.25 ± 0.30	12.4 ± 0.7	41.2 ± 2.1
SPI-1	5.10 ± 0.25	11.8 ± 0.6	38.5 ± 1.9
SPI-3	3.85 ± 0.20	10.2 ± 0.5	31.4 ± 1.6
SPI-5	3.60 ± 0.22	9.8 ± 0.6	29.7 ± 1.7

According to Table 3, the moisture-related characteristics of SPI films are significantly enhanced when MMT is added. There was a larger barrier effect with increasing clay levels, as the water vapor permeability fell steadily from 6.25 $g•mm/m^2•day•kPa$ in SPI-0 to 3.60 $g•mm/m^2•day•kPa$ in SPI-5. At 5% MMT, the water solubility declined from 41.2% to 29.7% and the moisture content dropped from 12.4% in the control film to 9.8%. The identical formulations achieved these reductions.

V. CONCLUSION

Biodegradable films made of Soy Protein Isolate-Montmorillonite (SPI-MMT) have been developed and characterized, and their potential as eco-friendly substitutes for plastic packaging has been demonstrated convincingly. Glycerol worked well as a plasticizer to keep the films flexible, and sodium-based MMT nanoclay improved the functional performance of the films by making them more stiff, enhancing their thermal stability, and adding barrier characteristics. The protein matrix and distributed clay nanolayers worked together to create a stronger, more compact structure. This helped overcome the problems with brittleness and high water vapor permeability that are inherent to pure SPI films. Given the importance of biodegradability, safety, and oxygen barrier properties in the food industry, the observed improvements suggest that SPI-MMT nanocomposites can be modified for a range of environmentally friendly packaging applications.

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