

Wheat Based Film

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Wheat is a grass plant of the Poaceae plants family; the scientific name of wheat plant is *Triticum*. Due to its mechanical and physical properties, wheat starch, gluten, and fiber are vital in the biopolymer industry. Glycerol as a plasticizer considerably increased the elongation and water vapor permeability of wheat films. Wheat fiber developed mechanical and thermal properties as a result of various matrices; wheat gluten is water insoluble, elastic, non-toxic, and biodegradable, making it useful in biocomposite materials.

wheat biocomposite

wheat starch

wheat gluten

wheat fiber

antioxidant

antimicrobial

1. Introduction

Plastic materials cause significant environmental damage and are one of humanity's greatest issues. Petroleum-based plastics are non-biodegradable, even after a hundred years. Plastic polymers, which are created from non-renewable elements, are one of the primary causes of global warming. Biocomposite materials are the ideal choice for possibly replacing fossil-based polymers. However, biocomposite materials require further development in terms of their characteristics ^[1].

Improving the properties of biocomposite material is still being investigated by researchers ^{[2][3][4][5][6][7]}. There is an abundance of research on wood and non-wood plants to extract starch, gluten and fiber in order to produce biocomposite materials. The ingredients of biocomposite materials are extracted from various types of agricultural crops, such as wheat, corn, cassava, hemp, jute, kenaf and other crops ^[8]. The advantages that make plants more useful than other sources for biopolymers are their availability, quality and quantity. In addition, plants offer variation in physical properties such as thickness, density, water content, water absorption and water solubility. There exists a variation in chemical constituents such as cellulose, hemicellulose, lignin and protein content in fiber, amylose and amylopectin ratio in starch ^[9]. Furthermore, their diversity in degree of polymerization, degree of crystallinity, water-vapor permeability and porosity make a difference in the biocomposite properties.

Wheat is a non-wood plants based fiber ^[10], which is planted in many countries and produces a lot of waste. Starch is the primary component of wheat, having a number of food and industrial applications ^[11]. In biocomposite application, wheat starch is used as biopolymer film with or without filler. Wheat fiber can be extracted from different parts of the plant to be used as reinforcement filler for either natural or synthesis matrix. Surface treatment is a method that is commonly used to clean, modify and improve the fiber surface to decrease surface tension and

to improve the interaction between the fiber filler and the starch film matrix or synthesis matrix [12][13][14][15][16]. Several publications have addressed the effects of sodium hydroxide treatment on the structure and properties of natural fibers such as kenaf, flax, jute, hemp, sugar palm and wheat fiber [17][18][19][20][21][22].

Straws such as wheat, rice and rapeseed straws, which known as cereal straws, are not only highly abundant but they are also a low-cost, potential candidate to be utilized in the development of green composites [23]. Wheat is one of the crops that is most sought after, and it is widely cultivated. The source of it comes from a grass named (*Triticum*) that is grown in countless countries around the entire globe. The total production of wheat in 2019–2020 was 763.9 million metric tons [24] and this percentage increases yearly.

One of the co-products from the starch and bioethanol industry is wheat gluten, which is utilized in many food and non-food application. It is widely used to develop films and other Bioplastics [25][26][27][28][29]. In 36 days, the decomposition of wheat gluten takes place in aerobic fermentation and takes 50 days in farmland soil without releasing any toxic residues into the environment [30]. Wheat gluten protein has a high decomposition rate, even when it is subjected to chemical and physical treatments. Therefore, wheat gluten polymer is a perfect alternative for the development of new biodegradable polymers, because of its decomposition properties and its unique viscoelastic and gas barrier properties [31]. Furthermore, wheat gluten has been explored as a raw material for non-food applications such as biopolymers [32][33][34]. In order to develop the eco-industry on our planet, biodegradable materials such as wheat-based biocomposites, which are distinguished with unique advantages such as, renewability, availability and low-cost raw materials.

Plasticizers used with starch to create the polymeric entangled phase, by reducing intramolecular hydrogen bonding [35][36][37]. Adding plasticizer to wheat starch improves the physical and mechanical properties because plasticizer increases the flexibility of the material. There are many types of plasticizer such as, fructose, sorbitol, urea and glycerol used to improve physical and mechanical properties. Similarly, to enhance mechanical and physical properties, plasticizers have been applied in many biocomposite materials, such as corn [38][39][40], sugar palm (*Arenga pinnata*) starch [41], cassava [42] and rice starch [43][44].

2. Wheat Plant

Wheat is a grass plant of the *Poaceae* plants family; the scientific name of wheat plant is *Triticum*. Wheat is one of the world's most ancient and essential cereal crops, which is grown across a wide range of climates and types of soils [45].

The main parts of the wheat plant are head spike, stem, leaves and roots. Wheat plants grow up to 2–4 feet tall. **Figure 1** shows wheat plants' main parts. The kernel of the wheat (also called the wheat berry) is the seed of the wheat plant [46], while the part that covers the kernel and protects it is called the beard; similar to all the grass plants, wheat plants stand on the stem. The leaves of wheat plants are long and comparatively thin; flag leaves are in the top of the leaves, which are responsible for the protection of the leaves. The nourishment from the soil to the plant comes through roots in the bottom of the plant [47].

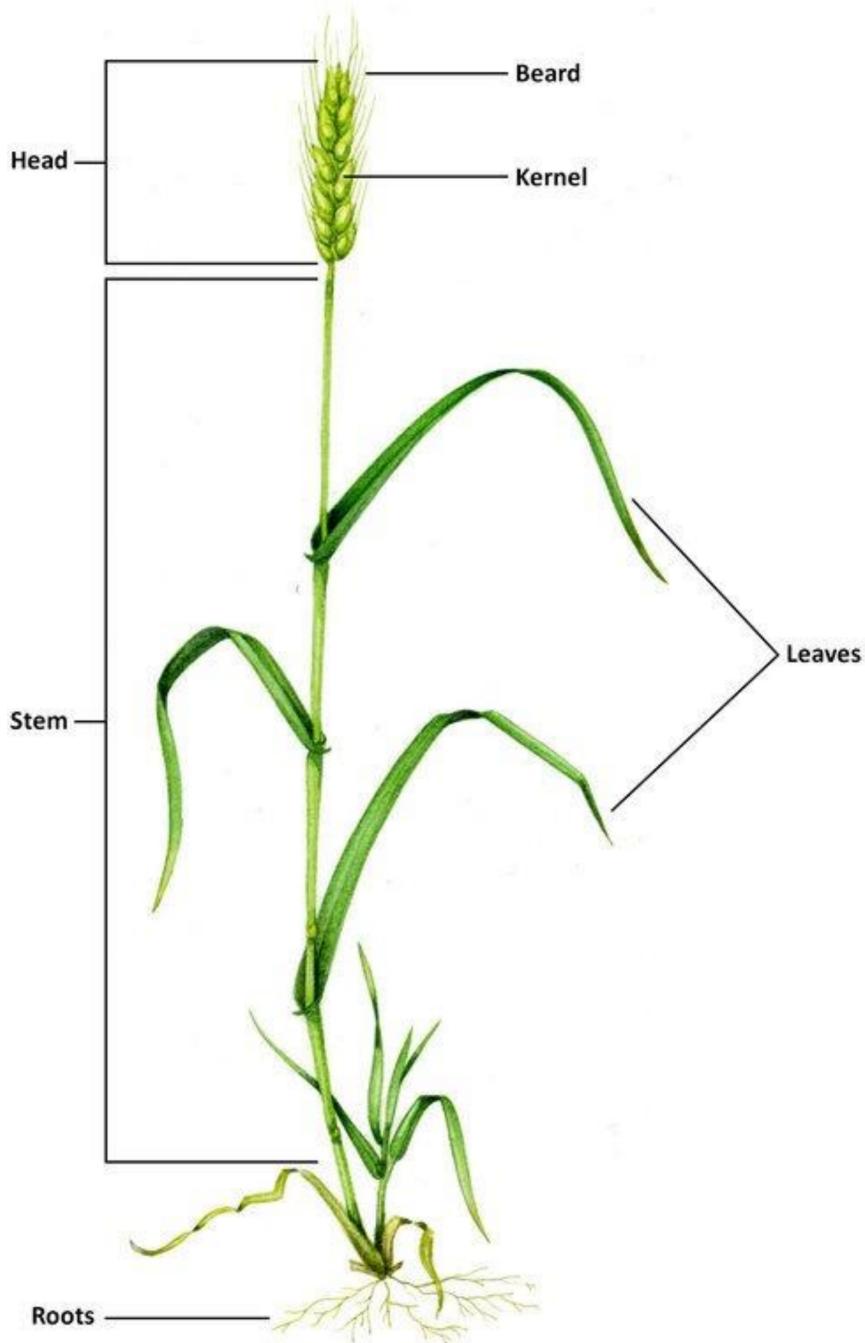


Figure 1. Wheat plant main parts [48].

3. Film Preparation and Properties Characterization of Wheat Starch Based Films

There are many factors that affect biopolymer properties, including: starch type, treatment temperature, additions such as plasticizer and co-biopolymers [35].

3.1. Physical and Chemical Properties of Wheat Starch

Wheat is one of the most widely farmed crops worldwide; the type of the soil and soil-dryness conditions affects the quality of the starch and other plant parts. The gelatinization enthalpy and swelling power of moderate soil-dryness treated starch are low. When compared to well-watered conditions, however, a greater gelatinization temperature, retrogradation enthalpy, and retrogradation percentage are found. According to Weiyang Zhang et al. [49], soil dryness affects amylose structure more than amylopectin structure in wheat grains. Furthermore, moderate soil dryness improves molecular structure and functional properties of the starch. **Table 1** shows a comparison between the chemical and physical structure of wheat, corn, rice and potato starches. There is no significant difference between the chemical composition of various starches.

The starch basically contains Amylose and Amylopectin. In biocomposite materials, it is important to identify the percentage of Amylose and Amylopectin, which directly affect the properties of the film or the matrix of the bio-polymer [50]. Amylose has a lower molecular weight than amylopectin; however, the high relative weight of Amylopectin reduces the mobility of polymer chains, resulting in high viscosity, whereas the linear structure of Amylose has demonstrated behavior more similar to that of conventional synthetic polymers [51]. The majority of natural starches are semicrystalline. Depending on the resource of the starch, the crystallinity of starch is around 20–45% percent. The short-branched chains in Amylopectin are mostly responsible for crystalline regain and appear as double helices with a length of around 5 nm. In the crystalline areas, the Amylopectin segments are all parallel to the big helix's axis [52]. Since proteins and polysaccharides are the primary components of natural polymers, the structure–property relationships in these materials are determined by their interactions with water and with each other in an aquatic medium [53].

Thianming Zhu et al. [54] applied different techniques to determine the percentage of Amylose in the starch; techniques included Differential Scanning Calorimetry (DSC), High-Performance Size-Exclusion Chromatography (HPSEC), iodine binding, and Megazyme amylose/amyopectin. Michael Ronoubigouwa Ambouroue Avaro [55] developed a method that used Tristimulus CIE Lab Values and developed a specific color board of Starch-iodine complex solution, the conversion of the regression values $L^*a^*b^*$ to Red, Green, Blue (RGB) values and to color hexadecimal codes. This method used a colorimeter device. A spectrophotometer is another device that can be used to detect the percentage of the Amylose by calculating the absorbent light that gets through the mixture of the starch and iodine solution [56][57][58].

Table 1. A comparison between the chemical composition and physical properties of wheat, corn, rice and potato starches [59][60][61][62][63][64][65][66][67][68][69][70][71][72].

Parameter	Type of Starch			
	Wheat Starch	Corn Starch	Rice Starch	Potato Starch
Amylose (%)	16.0–31.5	20.0–28	20–28	25–31

Parameter	Type of Starch			
	Wheat Starch	Corn Starch	Rice Starch	Potato Starch
Amylopectin (%)	68.5–75	75–83	65–85	76–83
Ash (%)	0.20–0.29	0.32–0.62	0.17–0.19	15.95–16.05
Proteins (%)	0.40–0.46	0.38–7.7	0.33–0.38	4.26–4.82
Density (g/cm ³)	1.5	1.356–1.4029	1.282	0.763
Moisture content (%)	10.65–13.3	10.45–10.82	3.60	15.98 ± 0.36

1. Omran, A.A.B.; Mohammed, A.A.B.A.; Sapuan, S.M.; Ilyas, R.A.; Asyraf, M.R.M.; Koloor, S.S.R.; Petrů, M. Micro- and Nanocellulose in Polymer Composite Materials: A Review. *Polymers* 2021, 13, 231.

2. Nurazza, N.; Asyraf, M.R.M.; Khalina, A.; Abdulkadir, A.; Sapuan, S.M.; Rafiqan, B.A.; Sabaruddin, F.A.; Kamarudin, S.H.; Nonnamin, M.N.P.; Ilyas, R.A.; et al. A Review on Natural Fiber Reinforced Polymer Composite for Bullet Proof and Ballistic Applications. *Polymers* 2021, 13, 646.

3. Alsubari, S.; Zuhri, M.Y.M.; Sapuan, S.M.; Ishak, M.R.; Ilyas, R.A.; Asyraf, M.R.M. Potential of Natural Fiber Reinforced Polymer Composites in Sandwich Structures: A Review on its Mechanical Properties. *Polymers* 2021, 13, 423.

4. Divya, Z.; Nidumaindin, R.; Selamat, M.; Ghazali, I.; Mohammad, N.; Huda, N.; Ilyas, R. A. Wheat Gluten: The primary protein in wheat grains [74]. It is mainly made from Wheat Gluten have potential to develop an edible film, adhesives, binders, and biomedical substances. The main advantages of wheat gluten films include being insoluble in water, elastic in nature, and non-toxic. Gluten matrix is biodegradable and glassy with characteristics similar to epoxy resin [75][76][77].

5. Mohd Nurazza, N.; Asyraf, M.R.M.; Khalina, A.; Abdullah, N.; Sabaruddin, F.A.; Kamarudin, S.H.; Ahmad, S.; Mahat, A.M.; Lee, C.L.; Aisyah, H.A.; et al. Fabrication, Functionalization, and Application of Carbon Nanotube-Reinforced Polymer Composite: An Overview. *Polymers* 2021, 13, 1047.

Wheat-gluten-based films can be produced via two common methods:

6. Ilyas, R.A.; Sapuan, S.M.; Harussani, M.M.; Hakimi, M.Y.A.Y.; Haziq, M.Z.M.; Atikah, M.S.N.; Asyraf, M.R.M.; Ishak, M.R.; Razman, M.R.; Nurazza, N.M.; et al. Polylactic Acid (PLA) Biocomposite: Processing, Additive Manufacturing and Advanced Applications. *Polymers* 2021, 13, 1326.

Wet-type mechanical milling is a common approach for producing nanoparticles for a variety of bio-materials, including starch and gluten [78]. For gluten, a milling process is used to obtain gluten powder. The wheat gluten suspension solution is made by mixing the gluten powder with ethanol (70% aqueous ethanol). Then fibers are

in Superplasticity. *Journal of Materials Processing Technology*, 2000, 103, 103-107.

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4.1.2 Dry Method

NATURAL FIBERS AIDS Res Hum Retrovir 1996; 12: 1427-1433

another gluten powder layer would be added through a sieve. These steps will be repeated until the desired thickness is achieved (2), by spreading dry powder on wet fiber in the mold. In this method, fiber must be wetted 9. Nam, S.; Netravali, A.N. **Green composites. I. physical properties of ramie fibers for environment-friendly green composites.** *Fibers Polym.* 2006, 7, 372-379.

again (after combing and drying), as the water will be a processing aid, after casting the gluten powder and wet fiber on the mold, the drying process needs to be conducted in a dryer oven ⁸⁰ composites: Sustainable and renewable green materials. *Renew. Sustain. Energy Rev.* 2017, **79**, 558–584.

Renewable green materials: Renew. Sustain. Energy Rev. 2017, 73, 558–584.

4.2 Properties Characterization of Films Based Wheat Gluten

11. Shevkani, K.; Singh, N.; Balaji, R.; Kaur, A. Wheat starch products

Due to the fact that polar amino acids such as glutamic acid, aspartic acid, lysine, arginine, serine, threonine, and

12. Liu, W.; Morsanty, A.R.; Askerzadeh, P.; Drza, L.T.; Misra, M. Influence of fiber surface treatment on

Amino acids contain reactive groups that can be useful in cross-linking and creating covalent connections, improving the mechanical characteristics of biocomposites [81]. It has been found that proteins rich in sulfur amino acids contain reactive groups that can be useful in cross-linking and creating covalent connections, improving the mechanical characteristics of biocomposites [81]. It has been found that proteins rich in sulfur amino acids

13. Bledzki, A.K.; Gassan, J. Composites reinforced with cellulose based fibers. *Prog. Polym. Sci.* 1999, 24, 221-274.

1999, 24, 221-274.
atoms in a single polypeptide molecule [82].

14. Norrrahim, M.N.F.; Huzalifah, M.R.M.; Farid, M.A.A.; Shazleeh, S.S.; Misehan, M.S.M.; Yasim-Antai, T.A.M.; Naveen, J.; Nurazmi, N.M.; Rani, M.S.A.; Harini, M.I.; et al. Greener Pre-treatment Approaches for Valorisation of Natural Fibre Biomass into Bioproducts. *Polymers* **2021**, *13*, 2023; higher water absorption, which reached approximately 520% after 210 min on cassava-starch-based films [42] and

295% after 240 min on corn-starch-based films [84]. All starch-based films showed very strong water absorption capacity. However, the amount of absorbed water is different from one starch to another. This behavior is attributed to the different crystalline structure of the starch.

Aisyah, H.A.; Norrrahim, M.N.F.; Ilyas, R.A.; Abdullah, N.; et al. Mechanical Performance and to the size of starch particles, the smaller the particles the earlier and higher water absorption. Also FTIR analysis . Applications of CNTs Reinforced Polymer Composites—A Review.. *Nanomaterials* 2021, **11**, 2186.

shows hydrogen bonded hydroxyl group peak more intensely with small-particle content compared to the larger 16C chain, this is explained by the Fitts-Petrie, iMyers and Atkinson capacity.⁸⁵ On the other hand, the hydroxyl peak of the 16C chain is more intense than the 18C chain, this is explained by the Fitts-Petrie, iMyers and Atkinson capacity.⁸⁵

Development Polymers **2021**, *13*, 2867. plasticizer reduces hydrogen bonding, which allows molecules to

move and increase the elongation while the high tensile appears when starch-starch hydrogen bonds overcomes 17. Sreenivasan, S.; Iyer, P.B.; Iyer, K.R.K. *Influence of delignification and alkali treatment on the fine starch-plasticizer bonds in a low amount of plasticizer* [87]. Reinforcing wheat-gluten with flax fiber improves the

10. Crossan, J.; Bledzki, A. K. Alkali Treatment of Jute Fibers: Relationship Between Tensile Strength and the elastic modulus, because of the hydrogen bonding between the fiber and the protein [80][88]

Heat treatment of wheat gluten at temperatures higher than 100°C reduces the effect of the reinforcing filler which reflected as reduction in the Young's modulus. This explains the reduction of wheat gluten adhesion

19. Jankel-Obermeier, I.; Sieber, V.; Paulstich, M.; Schneider, D. Solubilization of hemicellulose and lignin from wheat straw through microwave assisted alkali treatment. *Ind. Crops Prod.* 2012, 39, 92-93.

As confirmed by FTIR results, fiber chemical treatment removes lignin and hemicellulose and reduces the hydrophilic nature of the fiber and, hence, improves the interfacial adhesion between fiber and matrix [94][95].

20. Ilyas, R.; Sapuan, S.; Merati, M.S.; Asrofi, M.; Zainudin, E.S.; Rafiqah, S.; Aisyah, H. *Effect of wheat bran on the mechanical properties of wheat-based films*. *Int. J. Biol. Macromol.* 2011, **47**, 162–167.

21. Ilyas, R.A.; Sapuan, S.M.; Ishak, M.R.; Zainudin, E.S. *Sugar palm nanofibrillated cellulose required to cause chains breakdown. Although wheat-gluten-based films also prepared with solution cast method, (Arenga pinnata (Wurm.) Merr): Effect of cycles on their yield, physic-chemical, morphological compression molding have given better properties* [100]. The wheat-gluten films reinforced with fiber filler can be prepared either by wet or dry method:

22. Ahmad Ilyas, R.; Mohd Sapuan, S.; Ibrahim, R.; Abrah, H.; Ishak, M.R.; Zainudin, E.S.; Asrofi, M.; Tensile Strength of Wheat-Based Film Reinforced with Arenga pinnata Lignin: Effect of Lignin Content on the Mechanical Properties of Wheat-Based Film [101]. *Int. J. Biol. Macromol.* 2019, **123**, 379–388.

23. Zhao, L.; Xia, W.; Tarverdi, K.; Song, J. *Biocomposite boards from wheat straw without addition of bonding agent. Mater. Sci. Technol.* 2014, **30**, 603–610.

24. USDA. *World agricultural production. Ekon. APK* 2021. Available online: <https://www.fas.usda.gov/data/world-agricultural-production> (accessed on 14 August 2021).

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5. Antioxidant Properties of Wheat Based Film

26. Yang, Z.; Peng, H.; Wang, W.; Liu, T. *Crystallization behavior of poly(ϵ -caprolactone)/layered double hydroxide nanocomposites*. *J. Appl. Polym. Sci.* 2010, **116**, 2658–2667.

27. The inhibition of oxidation improves the stability of polymers to be effective in more applications [106]. The addition of crosslinking agent [107] and the use of the thermoplastic starch [108] to the wheat-based film [109] improves the mechanical properties of the film [110].

28. Guillet, J.; Mattozzi, A.; Johnson, E.; Hult, T.; Karlsson, M. *Transport and Tensile Properties of the resistance of plasticized Wheat-Gluten Film to Biodegradation* 2004, **15**, 2010–2026.

29. The constituents of polymers [74][109], the polymer type and the compound formulation and the end use application are governing the selection of the correct combination of antioxidants [110]. Wheat starch–chitosan films show the highest antioxidant capacity. However, the addition of α -tocopherol led to more heterogeneous film structure [111].

30. Feruloylated arabinoxylans extracted from wheat bran show high antioxidant activity in the presence of bound α -tocopherol [112].

31. Zárate-Ramírez, L.S.; Martínez, I.; Romero, A.; Partal, P.; Guerrero, A. *Wheat gluten-based materials plasticised with glycerol and water by thermoplastic mixing and thermomoulding*. *J. Sci. Food Agric.* 2011, **91**, 625–633.

6. Antimicrobial Properties of Wheat Based Film

32. Doménech, S.; Feuilloley, P.; Grataud, J.; Morel, M.-H.; Guilbert, S. *Biodegradability of wheat gluten-based bioplastics*. *Chemosphere* 2004, **54**, 551–559.

33. Antimicrobial property has received more attention recently especially in the bio-packaging food industry [113]. It has been found that composite wheat gluten-chitosan-based films can prevent microbial growth in intermediate-moisture conditions [114], where gluten is thought to act as an antimicrobial agents carrier [115][116][117]. Organic acids, enzymes, various plant extracts, bacteriocins, and essential oils have been integrated into biopolymers as antimicrobial agents [118][119][120].

34. Edwin, A.; Habeych, N. *Development of Starch-Based Materials*; Wageningen University: Wageningen, The Netherlands, 2009; ISBN 9789085854333.

35. Doménech, S.; Feuilloley, P.; Grataud, J.; Morel, M.-H.; Guilbert, S. *Development of Wheat Protein-Based Biodegradable and Biocompatible Polymers*. *Designing New Materials from Wheat Protein-Based Biodegradable Polymers* 2004, **5**, 1–121.

plants [124][126] have health benefits, antimicrobial and antioxidant properties [125][126]. (EOs) used to reinforce bio-matrix composites [127], such as reinforcing corn/wheat starch matrix with lemon oil, and the addition of lemon oil, significantly increased antimicrobial activity [128]. However, the addition of (EOs) concentration reduced the tensile strength, while the elongation at break does not change [129]. Potassium Sorbate (PS) has been used as an antimicrobial agent for wheat gluten films. (PS) shows antimicrobial activity, but it has been found that when the film is exposed to an absorbing medium, most of the PS is released [130]. Myrrh has been added as an antimicrobial to hydroxyethyl cellulose/wheat starch-based films and the results show the film kept the same mechanical properties of compression molded glassy wheat gluten bioplastics. *Ind. Crops Prod.* 2013, 44, 480–487.

33. Lagrain, B.; Goderis, B.; Brijs, K.; Delcour, J.A. Molecular Basis of Processing Wheat Gluten oil, significantly increased antimicrobial activity [128]. However, the addition of (EOs) concentration reduced the toward Biobased Materials. *Biomacromolecules* 2010, 11, 533–541.

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37. Kumari, N.; Bangar, S.P.; Petrů, M.; Ilyas, R.A.; Singh, A.; Kumar, P. Development and Characterization of Fenugreek Protein-Based Edible Film. *Foods* 2021, 10, 1976.

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40. Ibrahim, M.I.J.; Sapuan, S.M.; Zainudin, E.S.; Zuhri, M.Y.M. Preparation and characterization of cornhusk/sugar palm fiber reinforced Cornstarch-based hybrid composites. *J. Mater. Res. Technol.* 2020, 9, 200–211.

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