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Optimization of a production process for food bio-packaging based on cereal by-products

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Dedication

بسم الله الرحمن الرحيم، والحمد لله على جزيل نعمه وعظيم توفيقه، الحمد لله الذي هدانا لهذا وما كنا لنهتدي لولا أن هدانا الله،

وَأَمَّا بِنِعْمَةِ رَبِّكَ فَحَدِّثْ

أهدي هذا العمل المتواضع إلى والديّ العزيزين:

إلى أبي، سندي في هذه الحياة، الذي لا يمرّ يوم دون أن أكون ممتنًا لوجوده، وأفخر بأنه أبي، جزاه الله عني كل خير.

إلى أمي الحبيبة، التي ملأت حياتي حبًا وحنانًا، يا زهرةً تفوح بعطرها أينما حلّت، وكم أحمد الله الذي رزقني بك.

أهدي هذا العمل أيضًا إلى أشقائي وعائلتي الكريمة، الذين كانوا دومًا مصدر دعمٍ ومحبة.

إلى أصدقائي الأعزاء و (جماعة الوادي)، وزملائي الذين شاركوني هذا المشوار العلمي، وأخص بالذكر كل من تعرفت عليه خلال هذه الرحلة، فكنتم نعم الصحبة، ونعم الرفقة.

كما لا أنسى أن أرفع أسمى عبارات الشكر والامتنان إلى جميع أساتذتي الأجلاء، الذين علموني وربّوني في مختلف المراحل الدراسية.

وأخيرًا، أحمد الله تعالى على ما أنعم به عليّ، وأسأله أن يجعل هذا العمل خالصًا لوجهه الكريم، والصلاة والسلام على سيدنا محمد وعلى آله وصحبه أجمعين.

محمد الطاهر

Abstract

Theme: Optimization of a production process for food bio-packaging based on cereal by-products.

This study explores the development of biodegradable biofilms optimized per Plackett-Burman design and Response Surface Methodology (RSM) model from wheat bran starch as a sustainable alternative to conventional plastics. Starch was extracted from wheat bran with a yield of 23.14 ± 2.85% and used to produce bioplastic films with glycerol, gelatine, and citric acid. The films showed varying properties, with opacity ranging from 2.55 to 7.03, water solubility between 28.85% and 50.77%, and biodegradability in compost reaching up to 75%. Statistical analysis using Plackett–Burman design and Response Surface Methodology revealed that drying temperature and time had significant effects on biodegradability, while plasticizer type and citric acid amount were less influential. The RSM model showed a medium predictive performance, with R^2 of 81.53%, indicating a good fit with experimental data. These results confirm the potential of wheat bran as a valuable raw material for producing eco-friendly bioplastics.

Keywords: Wheat bran, Starch, Bioplastic, Plackett–Burman, RSM, Biodegradability, Solubility, Opacity.

Résumé

Thème : Optimisation d'un processus de production d'un bio-emballage alimentaire à base de sous-produits céréaliers.

Cette étude explore le développement de films bioplastiques biodégradables optimisés selon le plan Plackett-Burman et le modèle de la Méthodologie de Surface de Réponse (RSM), à partir de l'amidon de son de blé comme alternative durable aux plastiques conventionnels. L'amidon a été extrait du son de blé avec un rendement de 23,14 ± 2,85%, puis utilisé pour produire des films bioplastiques en combinaison avec du glycérol, de la gélatine et de l'acide citrique. Les films obtenus ont présenté des propriétés variables : une opacité allant de 2,55 à 7,03, une solubilité dans l'eau comprise entre 28,85% et 50,77%, et une biodégradabilité dans le compost atteignant jusqu'à 75%. L'analyse statistique, basée sur le plan Plackett-Burman et la RSM a révélé que la température de séchage et le temps de séchage ont un effet significatif sur la biodégradabilité, tandis que le type de plastifiant et la quantité d'acide citrique ont un effet moindre. Le modèle RSM a montré une performance prédictive satisfaisante avec un coefficient de détermination R^2 de 81,53%, indiquant une bonne adéquation avec les données expérimentales. Ces résultats confirment le potentiel du son de blé comme matière première prometteuse pour la production de bioplastiques écologiques

Mots-clés : Son de blé, Amidon, Bioplastique, Plackett–Burman, RSM, Biodégradabilité, Solubilité, Opacité.

ملخص

الموضوع: تحسين عملية إنتاج التغليف الحيوي للأغذية المصنوع من المنتجات الثانوية للحبوب.

تستكشف هذه الدراسة تطوير أغشية حيوية قابلة للتحلل الحيوي، مُحسّنة وفقًا لتصميم Plackett-Burman ونموذج RSM من نشاء نخالة القمح كبديل مستدام للبلاستيك التقليدي. تم استخراج النشاء من نخالة القمح بنسبة مردود بلغت 23.14 ± 2.85%، واستُخدم في إنتاج أفلام بلاستيكية حيوية باستخدام الجليسرول والجيلاتين وحمض الستريك. أظهرت هذه الاغشية الحيوية خصائص متفاوتة، حيث تراوحت العتامة بين 2.55 و7.03، والانحلالية في الماء بين 28.85% و50.77%، بينما وصلت نسبة التحلل الحيوي السماد العضوي إلى 75%. أظهرت التحاليل الإحصائية باستخدام تصميم Plackett-Burman ومنهجية سطح الاستجابة RSM أن درجة حرارة ومدة التجفيف كان لهما تأثير معنوي على قابلية التحلل الحيوي، في حين أن نوع المادة البلاستيكية وكمية حمض الستريك كان تأثيرهما أقل أهمية, حيث بلغ معامل التحديد $R^2 = 81.53\%$، مما يشير إلى توافق جيد مع البيانات التجريبية. تؤكد هذه النتائج الإمكانات الكبيرة لنخالة القمح كمادة خام مستدامة لإنتاج مواد بلاستيكية صديقة للبيئة.

الكلمات المفتاحية :نخالة القمح، النشاء، البلاستيك الحيوي، Plackett-Burman ، RSM، قابلية التحلل البيولوجي، الذوبان، العتامة.

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List of Abbreviation

Abbreviation	**Full Form**
RSM	Response Surface Methodology
CCD	Central Composite Design
ANOVA	Analysis of Variance
UV	Ultraviolet
PLA	Polylactic Acid
PHAs	Polyhydroxyalkanoates
PBAT	Polybutylene Adipate Terephthalate
PCL	Polycaprolactone
PE	Polyethylene
PET	Polyethylene Terephthalate
Bio-PE	Bio-based Polyethylene
Bio-PET	Bio-based Polyethylene Terephthalate
UNEP	United Nations Environment Programme
OECD	Organisation for Economic Co-operation and Development
SPSS	Statistical Package for the Social Sciences

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Introduction

Introduction

The world currently faces a critical environmental crisis due to the accumulation of petroleum-based plastics, which are non-biodegradable and persist in natural ecosystems for centuries. Approximately 19–23 million tonnes of plastic waste enter the environments each year, contributing significantly to pollution and creating long-term ecological damage (UNEP, 2021).

Bioplastics have emerged of the most viable solutions to this global challenge. These innovative materials, derived from renewable biomass sources, offering biodegradability and a significantly reduced carbon footprint as one throughout their lifecycle. Particularly promising are starch-based bioplastics, which combine widespread availability, cost-effectiveness, and compatibility with existing industrial processing methods (European Bioplastics, 2023).

A critical consideration in bioplastic development involves sourcing raw materials that are both sustainable and economically feasible, while avoiding competition with global food supplies. Wheat bran, an abundant byproduct of grain milling operations, presents an ideal solution as it contains substantial starch amount alongside other valuable functional components (Siraj, 2022). Despite these advantageous properties, the full potential of wheat bran for bioplastic applications remains largely untapped in current industrial practices (Dziki et *al.*, 2023).

Therefore, this research aims to optimize the production of biodegradable films from wheat bran starch, using the Plackett-Burman experimental design that combines appropriate factors followed by the response surface methodology (RSM), in order to obtain a better performing biodegradable biofilm with good transparency which is compared to standard bioplastic and commercial plastic.

Part I

Literature Review

Chapter 1

Food Bioplastics

CHAPTER 1: FOOD BIOPLASTICS

1.1. Definition of Bioplastics

Bioplastics are synthetic polymers that have completely or partly similar structural and functional characteristics from renewable natural materials such as crops, plants, or food waste. They can be molded into any shape for uses ranging from simple packaging to high-performance engineering parts. Unlike conventional plastics (fossil-based), bioplastics rely on renewable biomass sources, which have either biodegradable or non-biodegradable forms (OECD, 2013).

1.2. Origin and History of Bioplastics

According to Pathak et al. (2014), The development of plastics began in 1862 when Alexander Parkes introduced Parkesine, derived from cellulose and capable of being molded when heated and retaining its shape upon cooling. A few years later, in 1868, John Wesley Hyatt invented Celluloid, combining cellulose with alcoholized camphor. Celluloid became widely used in photography and film due to its flexibility and moldability. In 1907, Leo Baekeland revolutionized the plastics industry by synthesizing Bakelite, the first fully synthetic plastic made from phenol and formaldehyde, marking the beginning of modern plastics. The early 20th century also saw developments in casein-based plastics like Galalith and Erinoid, derived from milk protein and formaldehyde. Further breakthroughs came in the 1950s when Karl Ziegler and Giulio Natta developed polyethylene and polypropylene, respectively two of the most widely used plastics today earning them the 1963 Nobel Prize in Chemistry for their work on polymer synthesis.

1.3. Main Types of Bioplastics

A study by European Bioplastics (2023) shows that there are three broad bioplastics groups depending on their source material and degradability features (figure 1):

1.3.1. Bio-based but biodegradable

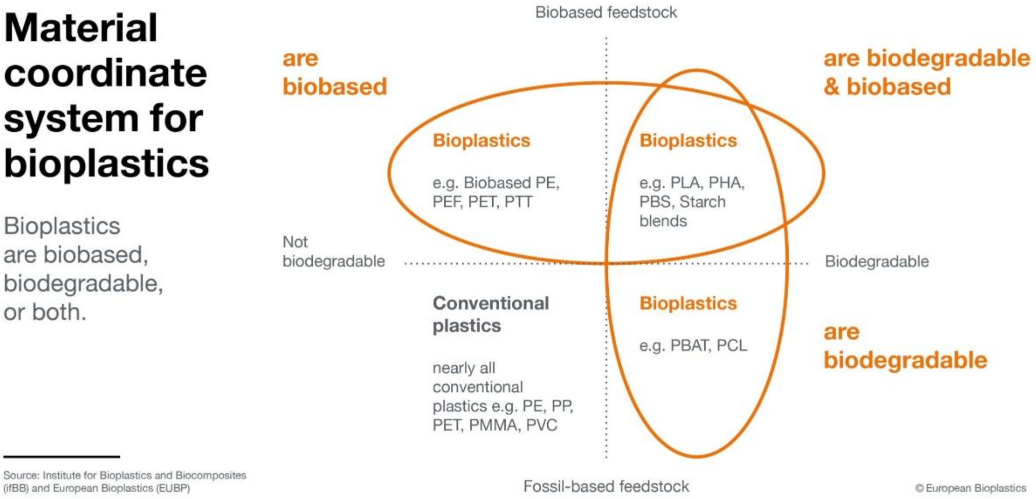
Polylactic acid (PLA), polyhydroxyalkanoates (PHAs), and starch blends which combine renewable content with complete biodegradability;

1.3.2. Bio-based but non-biodegradable

Like Bio-based Polyethylene (Bio-PE) and Bio-based Polyethylene Terephthalate (Bio-PET) that have plant-based feedstocks but compete with conventional plastics in terms of durability;

1.3.3. Fossil-based biodegradable polymers

Like PBAT and PCL that are oil-based but engineered to degrade. This kind of classification demonstrates that bioplastics are a series of sustainability choices, each one has different types serving either renewable feeding or end-of-life environmental performance. For comparison purposes, traditional plastics (Polyethylene (PE), Polypropylene (PP), PVC) neither have bio-based origin nor biodegradability (European Bioplastics, 2023).

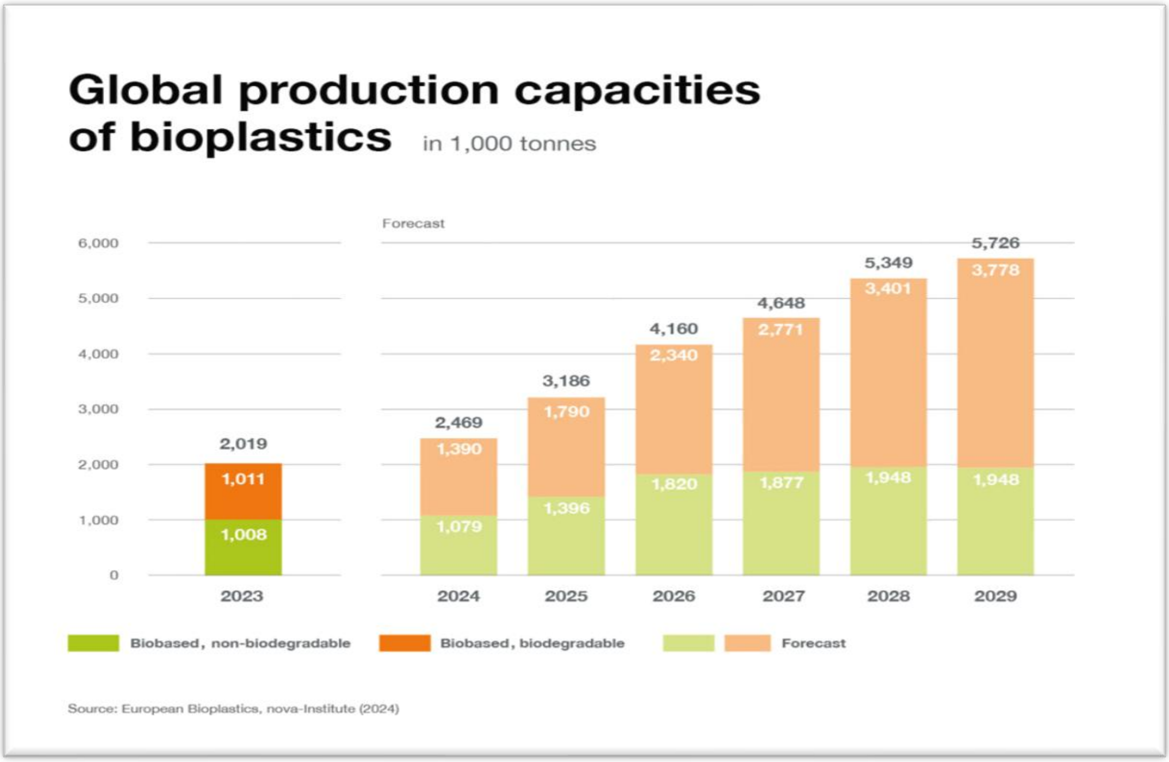


Source: *European Bioplastics (2023).*

Figure (01). Material coordinate system for bioplastic classification by origin and biodegradability.

1.4. Global Status of Bioplastic Production

In recent years, the global bioplastics industry has witnessed significant growth, though it still represents a small share of the overall plastics market. According to European Bioplastics (2024), the worldwide production capacity for bioplastics reached approximately 2.47 million tonnes, which accounts for less than 1% of total plastic production. However, the figure 03 projected to increase to around 5.73 million tonnes by 2029, indicating strong future potential. The packaging sector remains the dominant application area, comprising nearly 48% of the market. Geographically, Asia has become the leading production region, with more than 50% of global capacity, a share expected to surpass 70% in the coming years. Despite their growth, bioplastics require only 0.01% of global agricultural land for feedstock cultivation, minimizing competition with food crops. This expansion, driven by innovation in biodegradable polymers and sustainable practices, positions bioplastics as a promising alternative to conventional plastics.


Global production capacities of bioplastics in 1,000 tonnes

Source: European Bioplastics, nova-Institute (2024)

***Source:** European Bioplastics (2024).*

Figure (02). Global production capacities of bioplastic.

1.5. Properties of Bioplastics

Bioplastics have several characteristics such as light weight, biodegradability, and varying levels of strength and stability. Their properties depend on the raw materials used and affect how they perform in different applications.

1.5.1. Physical Properties

Thermobioplastics will normally exhibit desirable physical properties such as low weight, high clarity, and high surface gloss finish. PLA, for example, is transparent and glossy with a pleasing appearance for application in packaging. Rigidity and sensitivity to humidity can affect dimensional stability. Hydrophilic starch-based bioplastics absorb water and swell in humid conditions, hence limiting the structural integrity. The inclusion of hydrophobic fillers or crosslinking agents is typically taken into account to maintain minimal moisture sensitivity and enhance overall dimension and environmental stability (Beukelaer et al., 2022).

1.5.2. Mechanical Properties

Biobased and biodegradable thermoplastics such as PLA, PHAs, and starch-based polymers possess mechanical properties which, in some cases, compare with conventional petroleum-based plastics. In most cases, however, they are lower in impact strength and more prone to brittleness, particularly under dry conditions. The co-blending with plasticizers or blending with more flexible biopolymers is generally used to improve their ductility and toughness. PHAs, conversely, are highly versatile, and their mechanical performance can be controlled by adjusting their monomer composition. All of that aside, in most instances, further optimization is required to bring their mechanical ruggedness to the level of industrial plastics (Beukelaer et al., 2022).

1.5.3. Thermal Properties

Thermal properties are a big limitation for the majority of biodegradable polymers. For instance, PLA has a relatively low glass transition temperature of around 60 °C and has a melting point around 150-180 °C, making it unsuitable for hot usage. PHAs are relatively more heat-stable, though with differing performance depending on some structural characteristics. Generally, bioplastics will degrade or deform more easily when subjected to heat than traditional plastics, restricting their use in applications that involve exposure to heat, for example hot-fill packaging or microwave packaging. Thermal stability remains a subject of research (Beukelaer et *al.*, 2022).

1.5.4. Barrier Properties

Barrier performance is a significant factor in determining the suitability of bioplastics to packaging food and medicines. PLA demonstrates a favorable barrier against oil and grease, a moderate barrier to gases, but inferior resistance to water vapor and oxygen transmission compared to materials like Polyethylene Terephthalate (PET). PHAs and starch-based bioplastics have inconsistent barrier properties, which depend on the formulation. To improve them, manufacturers often use multilayer designs, special coatings, or add nanomaterials to reduce how easily gases and moisture pass through. Even with these improvements, enhancing the barrier performance of bioplastics is still a major challenge for using them in packaging that needs strong protection. (Beukelaer et *al.*, 2022).

1.5.5. Biodegradability

Biodegradability of the starch-based bioplastic films was tested using three different concentrations of the alpha-amylase enzyme: 0.25 mL, 0.5 mL, and 0.75 mL. At all enzyme concentrations, the raw bioplastic samples of all shared a similar and linear rate of degradation as time passes. However, nanosilica-reinforced bioplastics exhibited faster degradation within the first 24 hours because of the silicate layers that are supposed to enhance enzymatic activity. However, after this initial phase, the degradation rate of the reinforced samples decreased steadily and finally attained a comparable rate with that of raw bioplastics (Ashok et *al.*, 2019).

1.6. Uses and Applications of Bioplastics

1.6.1. Uses of Biobased, Non-Biodegradable Plastics

s Biobased, non-biodegradable plastics play a significant role in multiple industrial sectors where durability and long-term performance are essential. According to Rosenboom et al. (2022), these plastics are widely used in fibres, such as for textiles and non-woven materials, as well as in packaging, automotive, and electronic applications. Their chemical similarity to conventional plastics like PET and PE makes them ideal for applications that require both mechanical strength and chemical resistance. In the food industry, biobased alternatives like bio-PET and bio-PE are used in bottles, trays, and flexible films, offering compatibility with existing recycling systems and meeting food safety standards.

1.6.2. Applications of Biobased, Biodegradable Plastics

Biobased, biodegradable plastics are increasingly utilized in sectors prioritizing environmental sustainability and short product lifecycles. As Rosenboom et *al.* (2022) highlight, the packaging sector—including rigid and flexible formats—is the largest application area due to the demand for compostable alternatives. These materials are also gaining traction in agriculture, consumer goods, textiles, and even medical and hygiene products. In the food sector, they are used for compostable cutlery, produce bags, breathable films, and biodegradable containers, offering both functional and eco-friendly solutions. Despite progress, optimizing their barrier and thermal properties remains a research priority to broaden their use in more demanding applications.

1.7. Advantages and Disadvantages of Bioplastics

Bioplastic has many advantages and disadvantages, just like chemical plastics (Table 1). The pollution by plastic is a vital environmental problem, and it is driving the need for sustainable alternatives like bioplastics. They are produced using renewable resources (e.g., starch, cellulose, PHA, PLA). Bioplastics have advantages such as biodegradability, composability, and energy efficiency. They exhibit improved functional characteristics, including aroma barriers, moldability, and improved mechanical/thermal behavior compared to conventional plastics. Contrary to conventional plastics, which persist in ecosystems and are difficult to recycle, bioplastics mitigate environmental harm. While they are still problematic, their comparable or improved oxygen permeability, gas barrier, and water vapor transmission properties make them a viable, eco-friendly substitute. The move towards bioplastics is a step toward sustainability in accordance with global efforts to reduce pollution (Sidek et *al.*, 2019).

However, bioplastics also have some disadvantages. They are often more expensive to produce than conventional plastics. Some types are less heat-resistant and can be brittle, limiting their use in certain applications. Recycling is also a challenge, as not all bioplastics are compatible with current recycling systems. In some cases, their production may even require more energy. These drawbacks show that while bioplastics are more eco-friendly, they still need improvement to fully replace traditional plastics (Sidek et *al.*, 2019).

Table (01). Advantages and disadvantages of bioplastics compare to conventional plastics.

Types	**Advantages**	**Disadvantages**
Bioplastic	Sustainable	Costly
	Reduced Carbon Footprint	Thermal instability
	Reduce energy efficiency	Recycling problem
	Partly based on natural feedstock	Brittleness
Conventional plastic	Low cost	Based on petrochemical
	Good and excellent technical properties	Difficult to recycle
	Can save energy and resources	Mostly not biodegradable
	Thermal recycling possible	Uncontrolled combustion can release toxic substances

Source : *Sidek et al. (2010).*

Chapter 2

Valorization of wheat by-products

CHAPTER 2: VALORIZATION OF WHEAT BY-PRODUCTS

2.1 Overview of wheat

2.1.1. Definition and origin

Wheat (*Triticum aestivum*) is a globally significant cereal crop with a complex hexaploid genome, which arose from ancient hybridization events. Its genetic structure contains three sets of homoeologous chromosomes, providing redundancy that can mask genetic variation but also offers unique opportunities for trait improvement. With the aid of modern genomic tools and high-throughput phenotyping, researchers and breeders are now better equipped to identify and modify genes that control important traits like yield, stress tolerance, and grain quality. These advances are critical for developing wheat varieties that can meet future agricultural demands in the face of environmental and population pressures (Borrill et *al.*, 2019).

The origin of wheat dates back more than 10,000 years to the Fertile Crescent, a region encompassing parts of modern-day Iraq, Syria, Turkey, and Iran. It was first domesticated from wild grasses such as (einkorn) and (emmer) wheat by Neolithic farmers. This early domestication marked a turning point in human history, leading to the rise of settled agriculture and the development of early civilizations. From its origin in the Near East, wheat gradually spread to Europe, Africa, and Asia, becoming a central crop in many ancient cultures and remaining vital to global agriculture today (Shewry & Hey, 2015).

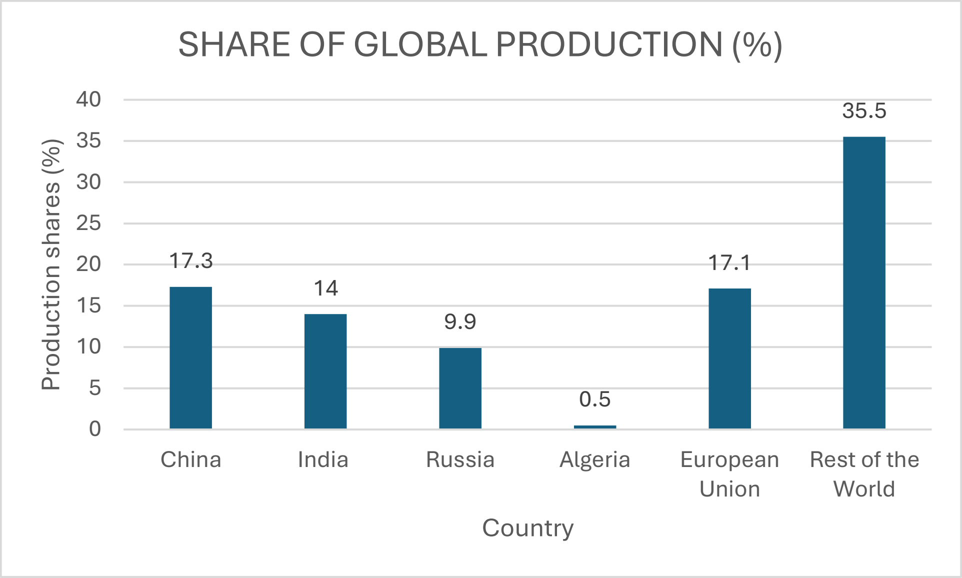
2.1.2. Classification of wheat

Botanically, wheat belongs to the genus *Triticum* within the grass family Poaceae. The most widely cultivated species is *Triticum aestivum* L., a hexaploid that dominates global wheat production. Another key species is *Triticum durum Desf.*, used mainly for pasta products. Additionally, ancient species like *Triticum spelta*, *Triticum dicoccum* (emmer), and *Triticum monococcum* (einkorn) are still grown on a limited scale. These species differ in their genetic makeup and agronomic properties, which are significant in breeding and wheat improvement efforts (Shewry & Hey, 2015).

2.1.3. Global Production and Economic Relevance

Wheat is the second most produced cereal crop globally, following maize, and it holds the highest volume of global trade among all agricultural products. During the 2023–2024 marketing year, worldwide wheat production was estimated at 785 million tons. China, India, and Russia were the top producers, jointly contributing around 41% of global output. Algeria contributing of approximately 3.9 million tons. The European Union, if considered as a single entity, produced more wheat than any individual country except China, highlighting its major role in global wheat supply (figure 03) (World Population Review, 2024).

Moreover, wheat markets are increasingly influenced by climate variability, rising input costs, and geopolitical factors, which affect production, pricing, and trade flows. For instance, regional conflicts or extreme weather events have led to supply chain disruptions and price volatility in recent years (FAO, 2023).



Source: *World Population Review (2024).*

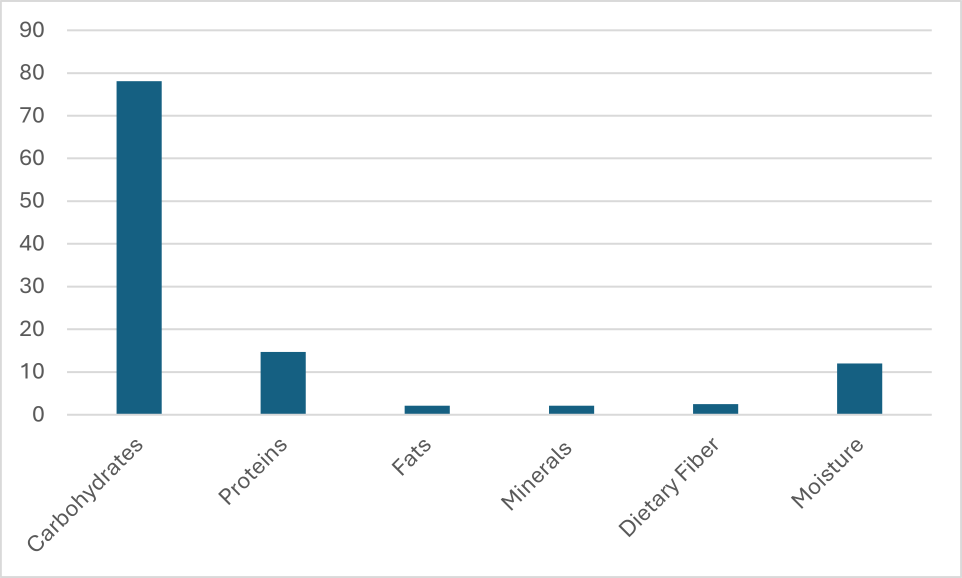
Figure (03). Production shares of wheat by region.

2.1.4. Chemical composition and nutritional importance of wheat

2.1.4.a. Chemical composition of wheat

Wheat grain is chemically composed of various macro- and micronutrients essential for both human health and industrial applications (figure 04). The major component of wheat is carbohydrate, making up approximately 78.10% of its dry weight, in the form of starch. Protein content is relatively high among cereals, averaging around 14.70%, which includes gliadin and glutenin (Shewry & Halford, 2002). Lipid content remains low, around 2.10%, wheat contains about 2.10% minerals, such as iron, zinc, magnesium, and selenium (Kumar et *al.*, 2011).

Wheat also contains dietary fiber, enzymes, phenolic compounds, and bioactive molecules such as alkylresorcinols and phytosterols, which contribute to its functional and nutritional value (Zhao et *al.*, 2021). B vitamins like thiamine, niacin, and folate are found predominantly in the bran and germ, making whole wheat products more nutritionally rich compared to refined flour.



Source: *(Kumar et al., 2011).*

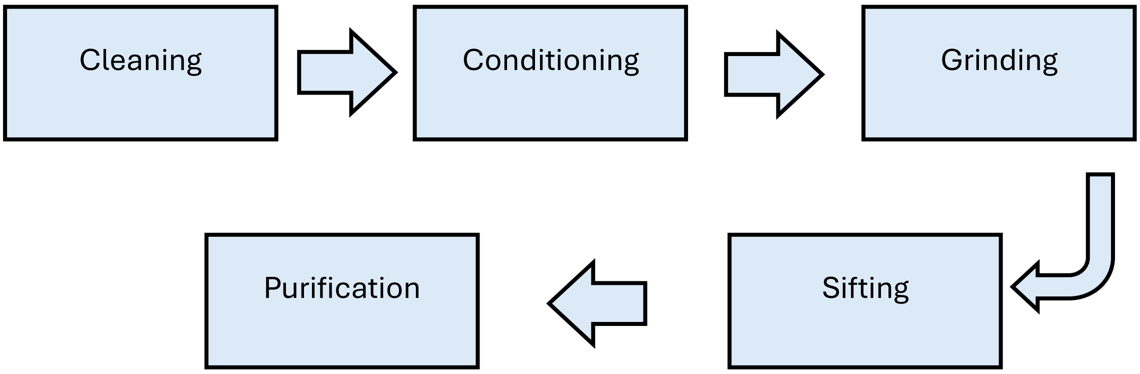
Figure (04). Chemical Composition of Wheat Grain

2.1.4.b. Nutritional importance of wheat

Wheat provides nearly 55% of carbohydrates and 20% of food calories, particularly in less developed countries where bread, noodles, and other products may provide a substantial proportion of the diet. It contains carbohydrate 78.10%, protein 14.70%, fat 2.10%, minerals 2.10%, and considerable proportions of vitamins (thiamine and vitamin B) and minerals (zinc and iron). Wheat is also a good source of trace minerals like selenium and magnesium (Kumar et *al.*, 2011).

2.1.5. Wheat Processing and Milling

In order to produce flour and other by-products, wheat must be processed through a number of precise steps that separate the endosperm from the bran and germ (figure 05). Cleaning is the first step in the process, where contaminants like dust, stones, and foreign seeds are eliminated using sophisticated methods like color-sorting and debranning. The next step is conditioning, also known as tempering, which involves bringing the grain's moisture content down to about 14–16% in order to toughen the bran and soften the endosperm, which makes separation easier. In order to separate the flour from the outer layers, the grain is gradually broken down during the grinding phase using roller mills. Particles are then sorted by size using sifting, producing refined flour and by-products like germ, bran, and shorts. During the procedure, quality control tools like Near-Infrared (NIR) spectroscopy, Single Kernel Characterization Systems, and digital grain imaging ensure optimal flour yield and product consistency (Dziki et *al.*, 2023).



***Source**: Dziki et al. (2023).*

Figure (05). Modern Wheat Milling Process

2.2. Wheat by-products

2.2.1. Classification of wheat milling by-products

Wheat milling generates various by-products that account for approximately 25-30% of the total grain weight (Huang et *al.*, 2012). These by-products primarily wheat bran, middling's, shorts, red dog, and feed flour play crucial roles in food, feed, and industrial sectors.

2.2.1.a. Wheat Bran

The outermost layer of the grain, is rich in non-starch polysaccharides (arabinoxylans and cellulose), comprising up to 60% of its dry weight. It also contains significant amounts of protein (14-20%), starch (11-24%), and minerals (3-8%). Bran is extensively used in functional foods, whole-grain bakery products, and animal feed due to its high fiber and antioxidant content. However, stabilization is necessary to prevent rancidity due to its lipid content (Hemery et al., 2007).

2.2.1.b. Middlings

Middlings are composed of small particles from the bran, germ, and endosperm. Rich in protein (15–18%), fat (up to 5%), and fiber (7–9%), they are widely used in livestock diets and increasingly in biofuel production. They offer up to 96% of the digestible energy of corn, making them a cost-efficient component in animal feed (Huang et *al.*, 2012)

2.2.1.c. Shorts

Wheat shorts are a fine by-product from wheat milling, primarily consisting of endosperm with small amounts of bran and germ. According to Huang et *al.* (2012), wheat shorts are rich in digestible energy and nutrients, including starch and amino acids, making them highly suitable for use in swine diets. Their relatively low fiber content and fine particle size enhance feed processing efficiency, especially in pelleted formulations. The study demonstrated that wheat shorts offer considerable nutritional value and can serve as a cost-effective component in livestock feeding programs.

2.2.1.d. Red dog

Red dog is a fine, powdery by-product of wheat milling that contains parts of the aleurone layer, flour, and small bits of bran and germ. It is high in starch (around 43%), low in fiber, and has good protein content, making it a valuable energy source in animal feeds, especially for pigs and poultry (Casas et *al.*, 2017).

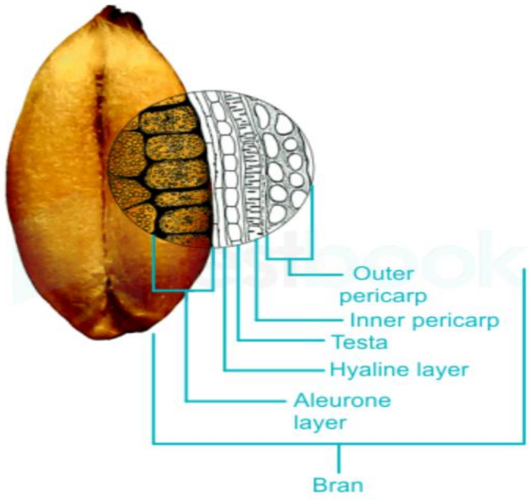
2.2.2. Valorization and Economic Potential of Wheat By-products

Innovative approaches are redefining the use of wheat milling by-products into high-value applications with both nutritional and economic significance. Wheat bran, once considered waste, is now processed to extract dietary fibers, phenolic antioxidants, and phytochemicals that are incorporated into functional foods, nutraceuticals, and pharmaceutical products. Wheat germ is similarly valorized through the extraction of oil and protein rich in essential fatty acids and vitamins, used in health and sports nutrition formulations as well as in dietary supplements and cosmetics due to its skin-nourishing properties (Siraj, 2022). Furthermore, wheat middlings are increasingly utilized in biocomposites, bioethanol production, and animal feed, offering a cost-efficient source of digestible nutrients that reduce feeding costs without compromising livestock performance (Casas et *al.*, 2018). Shorts and red dog also serve as effective fermentation substrates for producing enzymes and organic acids, contributing to industrial biotechnology and circular economy goals (Danciu et *al.*, 2023). These diverse applications not only enhance the economic value of wheat by-products but also promote more sustainable and efficient agro-industrial systems.

2.3. Overview of wheat bran

2.3.1. Definition and structure

Wheat bran is the outermost layer of the wheat grain, derived as a by-product during the milling process. It represents a significant source of functional ingredients (figure 06), with applications in both food and non-food industries (Rosa et *al.*, 2013). Rich in dietary fiber, B vitamins, minerals, and bioactive compounds, wheat bran has gained increasing attention for its health-promoting properties. Global trends show a marked rise in the use of wheat bran in food products, growing from just 52 products in 2001 to around 800 by 2011 (Preuckler et *al.*, 2013).



Source: *Mateo Anson et al. (2012)*

Figure (06). Structure of wheat bran layers**.**

2.3.2. Extraction of wheat bran

Bran constitutes up to 13–19% of total wheat grain weight depending on whether its dry or wet process of extraction (Hossain et *al.*, 2013). In dry milling, bran is separated from the endosperm which is then ground into flour. Wheat grains are conditioned by adding water to reach about 15% moisture before milling, then left to rest in tempering bins. Tempering time varies based on the grain's hardness. During tempering, water softens the endosperm and loosens the pericarp and germ, causing them to separate easily. Conditioned grains pass through corrugated metal rollers, which crack the kernels and enable separation into endosperm, germ, and bran (Onipe et *al.*, 2015).

2.3.3. Traditional and emerging uses of wheat bran

2.3.3.a. Traditional uses

Wheat bran has been utilized as a low price and wholesome animal feed. It is frequently fed to dairy cows, sheep, and poultry and offers important fiber, protein, and minerals (Zhang et *al.*, 2022). in addition, wheat bran can be used as a source of bioenergy. Because of its high fiber and carbohydrate content, it can be used in anaerobic digestion or microbial fermentation to produce bioethanol and biogas (Saini et *al.*, 2017).

2.3.3.b. Emerging uses

Recently, the food and industrial sectors have become more interested in wheat bran. It is increasingly added to bakery and breakfast cereal products to boost fiber content and improve health benefits (Preuckler et *al.*, 2013). Its application in biodegradable materials has also been researched. Wheat bran, for instance, is now utilized as a filler in bioplastic composites, such as blends of polylactic acid (PLA) and PBSA, where it improves environmental sustainability and lessens plasticizer migration (Sanyang et *al.*, 2022). These advancements demonstrate that wheat bran is evolving into a useful component in the food and materials industries, no longer merely a by-product.

2.3.4. Physico-chemical properties relevant to valorization

The composition is significantly influenced by wheat variety, cultivation conditions, and the milling methods used especially those that determine how much endosperm or starch remains attached to the bran during separation (Raghu Babu et *al.*, 2018).

Chemically, wheat bran consists of both soluble and insoluble dietary fibers, including arabinoxylans (19-25%), cellulose, hemicellulose, lignin (~3%), and resistant starch (17-29%). It also contains 14-18% protein, with a well-balanced amino acid profile—particularly high in lysine—and about 0.5% lipids. The bran is also enriched with essential minerals like iron, zinc, manganese, magnesium, and phosphorus, although 80% of phosphorus is bound in the form of phytates, which may reduce the bioavailability of other minerals due to complex formation (Raghu Babu et *al.*, 2018).

Each of the several structural layers that make up wheat bran adds to its nutritional content. The outermost layer, or pericarp, which accounts for around 5% of the grain's weight, is abundant in phenolic compounds, xylans, cellulose (~20%), and lignified cell walls. Alkylresorcinols, which are recognized for their antioxidant and perhaps anticancer qualities, are found in the testa, or seed coat. Important substances like ferulic acid and arabinoxylan are found in the hyaline layer, or nucellar tissue. With over 80% of the grain's niacin, 60% of its vitamin B6, and 32% of its thiamine, as well as substantial levels of lignans, antioxidants, phytic acid, and other bioactive compounds, the aleurone layer is especially nutrient-dense. All things considered, wheat bran has 34–63% dietary fiber, which promotes gut health, enhances satiety, bulks up the stool, and helps avoid diarrhea (Raghu Babu et *al.*, 2018).

2.3.5. Applications in bioplastic production

According to Rossi et *al.* (2023), wheat bran, being abundant and rich in fiber, can be effectively valorized as a natural filler in bio-based polymers, helping to improve environmental sustainability while reducing material costs. When incorporated into polymer matrices, such as biodegradable plastics, wheat bran contributes to improved biodegradability, reduced plasticizer migration, and enhanced mechanical performance under certain conditions. Similarly, Majewski and Cunha (2022) evaluated the use of wheat bran as a natural filler in polymer processing and found that its fine particle size and compatibility with polymers like polyethylene (PE) and polylactic acid (PLA) made it suitable for composite manufacturing. Their research showed that adding wheat bran influenced the physical, thermal, and structural properties of the materials, making it a promising alternative to synthetic fillers in developing eco-friendly plastics.

Part II
Experimental Study

Chapter 1

Materials and Methods

CHAPTER 1: MATERIALS AND METHODS

This work was carried out in the pedagogic laboratories of the Agricultural Sciences Department at Laghouat University, with the aim of optimizing the manufacturing process of bioplastic based on wheat bran starch. This process involved two stages: extraction of the starch and production of a biofilm.

1.1 Process of Starch Isolation from Wheat Bran

1.1.1. Sample preparation

A large quantity of wheat bran was obtained from a local source in Laghouat region. Initially, the bran was sieved using a 2.2 mm mesh to remove visible impurities such as insects and large particles.

1.1.2. Starch extraction process

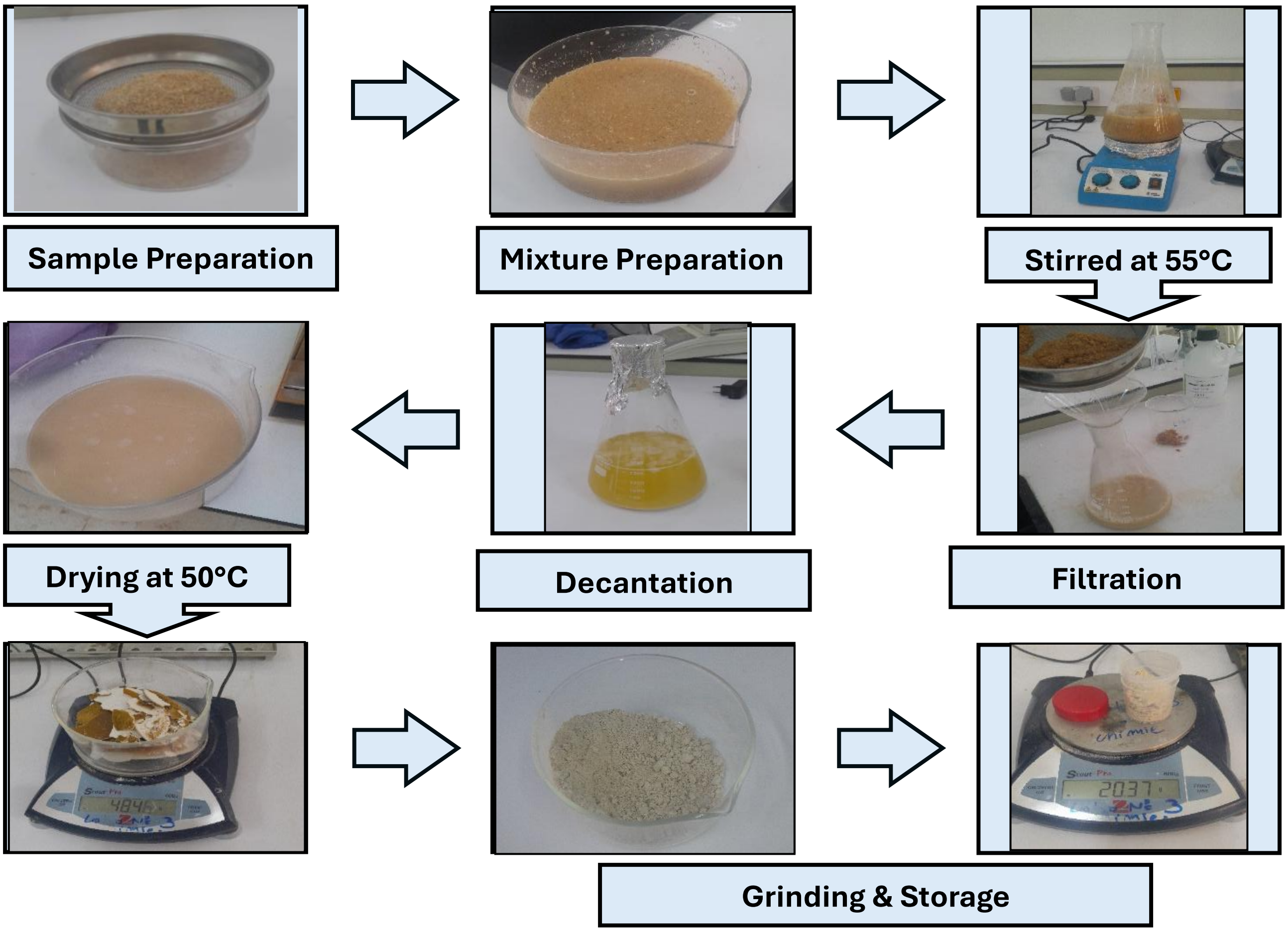
To isolate starch from wheat bran, the protocol of Sardari et al. (2019) with some modifications was used. In 2L Erlenmeyer flask, 100 g of prepared wheat bran was added to 1 L of distilled water (ratio of 1:10). The mixture was then stirred using a hot plate magnetic stirrer at 55 °C for 4 hours to facilitate starch release. The resulting suspension was filtered using a 200 µm sieve placed over a funnel into another 2 L Erlenmeyer flask. The remaining solid residues were rinsed with 500 mL of distilled water and then filtered again into the Erlenmeyer to recover additional starch. The collected suspension was left undisturbed for approximately 2 hours or longer to allow the precipitation of starch in the bottom. Once decantation was sufficient, the supernatant was gently collected into a beaker without disturbing and losing the settled starch. The recovered supernatant was left to stand for another day to recover as much starch as possible. The precipitated starch was poured into glass Petri dishes and placed in a drying oven set at 50 °C and left overnight for complete dehydration. The dried starch was then ground into a fine powder using an electric grinder and stored in clean plastic vials for further use.

1.1.3. Yield Calculation

The initial weight of each empty dish and dish with fresh starch were recorded to allow later determination of starch yield. After drying, the Petri dishes were weighed again, and the weight difference was recorded. The starch yield was expressed as a percentage (w/w) and calculated for each replicate using the following formula:

$$\textbf{Yield}\ (\%) = \frac{\mathbf{P2}}{\mathbf{P1}} \times \mathbf{100}$$

P1: initial weight of wheat bran; **P2:** weight of recovered and dried starch.



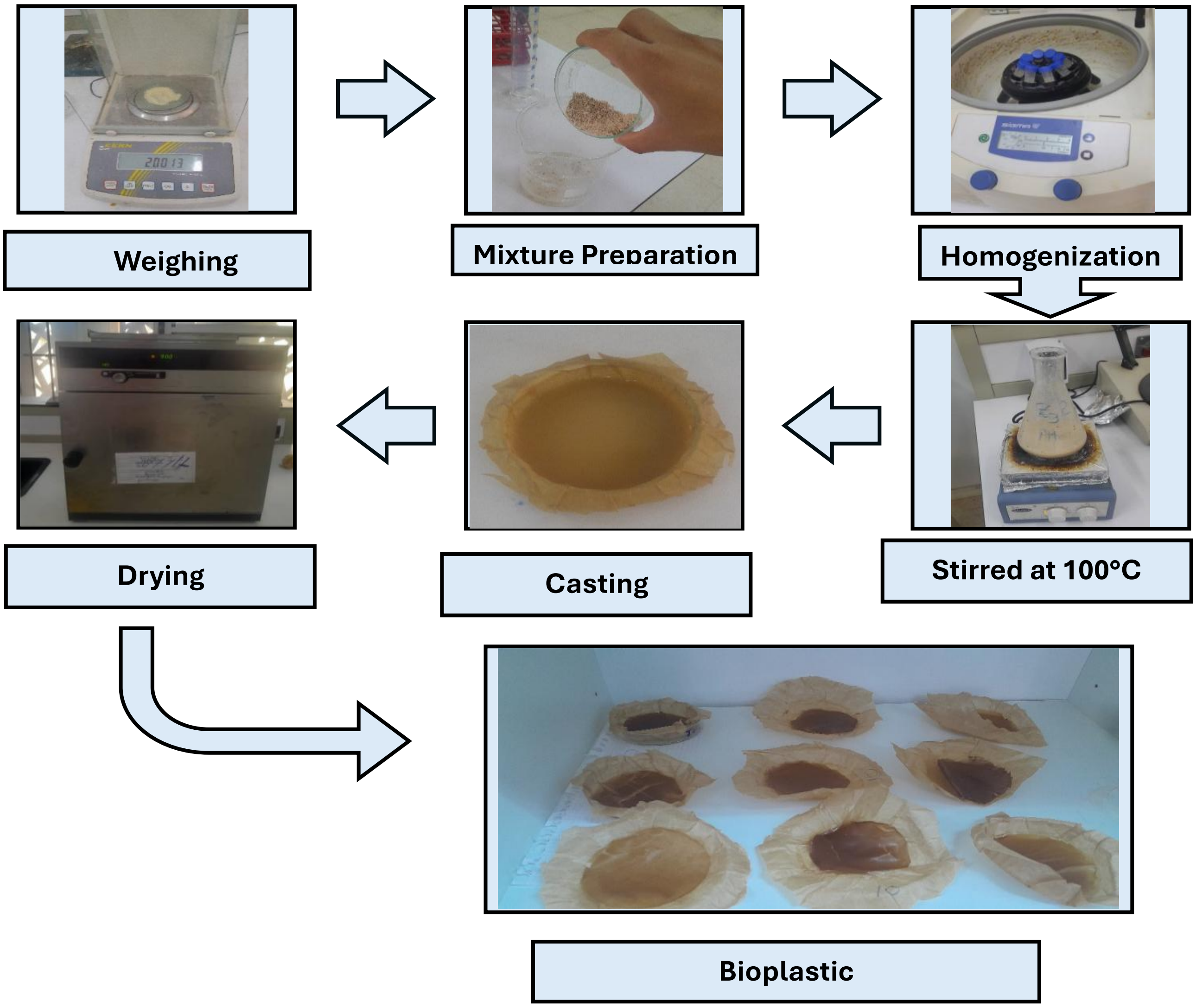
Source: *Original (2025).*

Figure (07). Starch extraction process from wheat bran.

1.2. Bioplastic Production under Standard Conditions

The preparation of bioplastics was carried out according to Marichelvam et al. (2019), with several modifications.

In 250 ml beaker, 10 grams of starch were introduced and dissolved in 100 mL of distilled water, maintaining a 1:10 starch-to-water ratio. Subsequently, 3 grams of glycerol, 2 grams of gelatin, and 1 gram of citric acid were added to the solution. The mixture was centrifuged at 180 rpm for 10 minutes to ensure uniform distribution of all components. This step facilitated solubilization and pre-gelatinization of the starch. After centrifugation, the mixture was heated on a hot plate at 100 °C, and manual stirring was done for 70 min. The mixture was then poured uniformly into glass Petri dishes lined with parchment paper lightly coated with edible oil to prevent adhesion and facilitate demolding, then transferred to a drying oven and incubated at 90°C for 2 hours. Following oven drying, the Petri dishes were removed and the bioplastic films were left to air-dry at ambient temperature for 2–3 days The final bioplastic films were then carefully removed and stored at room temperature until further analysis.



Source: *Original (2025).*

Figure (8). Production of Bioplastic under Standard Conditions.

1.3. Bioplastic Production According to the Plackett-Burman Experimental Design

Table (02). Factors used in the Plackett-Burman experimental design

Factor	**Level (−1)**	**Level (+1)**
Plasticizer type	Glycerol(3g) +Gelatin (2g)	Glycerol only
Quantity of citric acid	0.5 g	2 g
Drying temperature	70 °C	110 °C
Drying time	1 hour	3 hours

To optimize the bioplastic production process using starch extracted from wheat bran, the Plackett-Burman experimental design (Plackett-Burman 1946) were applied. This design was selected for its efficiency in screening multiple variables simultaneously with a limited number of experiments. It allowed us to identify and screen the most influential factors affecting the quality and integrity of the resulting bioplastic films.

Table (03). Experimental Matrix of the Plackett-Burman Design

Trial Order	Glycerol	Acid Qantity (g)	Drying Temperature (°C)	Drying Time (h)
1	-1	-1	1	1
2	1	1	-1	1
3	1	-1	1	1
4	-1	1	1	-1
5	-1	-1	-1	-1
6	-1	1	1	1
7	1	-1	1	-1
8	1	1	1	1
9	-1	-1	-1	1
10	1	1	1	-1
11	1	-1	-1	-1
12	-1	1	-1	-1

In our case, the primary goal of this experimental design was to determine the significant effects of four factors (plasticizer type, quantity of citric acid, drying temperature, drying time) on the properties of the bioplastic elaborated (Table 02).

According to Table 03, the Plackett-Burman matrix showed the presence of 12 trials, testing each factor at two levels, coded −1 (low) and +1 (high).

1.4. Properties of elaborated bioplastics

1.4.1. Biodegradability

The biodegradability of the bioplastic was determined according to Marichelvam et *al.* (2019) with some modification. Samples were cut into 2 cm × 2 cm squares and weighed to record the initial mass. Each sample was then buried 3 cm deep in a plastic cup filled with locally sourced compost soil. After 15 days of incubation at ambient conditions, the samples were carefully removed, cleaned to remove any adhering soil, and weighed again to determine the final mass. The biodegradability percentage was calculated using the following equation:

$$\textbf{Biodegradability}\ (\%) = \frac{\boldsymbol{W_i - W_F}}{\boldsymbol{W_i}} \times \mathbf{100}$$

Where: $\boldsymbol{W_i}$ is the initial weight; $\boldsymbol{W_F}$ is the final weight

Source: *Original (2025).*

Figure (09). Biodegradability test in compost

1.4.2. Water solubility

The water solubility of the biofilms was determined according to the method described by Rhim et al. (2005). To determine the initial dry mass (DM), samples from each biofilm were first dried in an oven at 110 °C for 6 hours. Each dried sample was then placed in a separate beaker containing 40 mL of distilled water. The beakers were sealed with aluminum foil and kept at room temperature for 24 hours with slight agitation. After this period, the remaining pieces of the biofilms were removed, rinsed with distilled water, and dried again in the oven at 110 °C for another 6 hours to determine the mass of the undissolved material.

The mass of the water-soluble portion (MS) was calculated by subtracting the undissolved dry mass from the initial dry mass. The water solubility rate (WS%) of the biofilm was then calculated using the following formula:

$$\mathbf{WS}\ \% = \frac{\boldsymbol{MS}}{\boldsymbol{DM}} \times \mathbf{100}$$

1.4.3. Opacity

The opacity of the bioplastic samples was evaluated following a modified method based on a previous study of Asfaw et *al.* (2022). The films were cut into rectangular strips measuring 0.8 cm × 3 cm. The thickness of each film was determined using a caliper. Each specimen was then placed directly in the cuvette of a spectrophotometer. Light transmittance was measured at a wavelength of 500 nm. The opacity was calculated using the formula, where A500 is the absorbance at 500 nm and X is the thickness in mm. the value was reported for each sample:

$$\mathbf{Opacity} = \frac{\boldsymbol{A}_{\mathbf{500}}}{\mathbf{X}}$$

Source: *Original (2025).*

Figure (10). Measurement of opacity by UV-Visible spectrophotometry.

1.5. Optimization of Bioplastic Production Using Response Surface Methodology (RSM)

According to our main objective of producing a biodegradable biofilm, the optimization analysis by a response surface methodology (RSM) was continued, which biodegradability was considered as the studied response.

Table (04). Central Composite Design Matrix Used for RSM Optimization (RSM-CCD)

Trial Order	Drying temperature	Drying time
1	0.000	0.000
2	0.000	0.000
3	-1.000	1.000
4	1.414	0.000
5	0.000	-1.414
6	1.000	1.000
7	-1.414	0.000
8	-1.000	-1.000
9	0.000	1.414
10	0.000	0.000
11	0.000	0.000
12	1.000	-1.000
13	0.000	0.000

Following the analysis of the Plackett–Burman design, drying temperature and drying time were identified as the most significant variables affecting the biodegradability in compost. In order to optimize this parameter, a second experimental design was developed using Response Surface Methodology (RSM) based on a Central Composite Design (CCD). When it was used to model the connections between these two variables and the response measured (biodegradability) (Myers, Montgomery, & Anderson-Cook, 2016).

An augmented matrix of 13 experimental runs was generated and applied. For each run, bioplastic films were prepared under the defined conditions using the optimized base formulation derived from the Plackett–Burman screening phase. All other components (starch source, plasticizer type, and citric acid concentration) were kept constant (table 04).

After films formation according the 13 trials established by the surface response plan (RSM), the Biodegradability bioplastics was evaluated following a method previously explained (But only for 10 days due to time constrain).

Data Processing

All optimization analyses (create of Plackett-Burmen matrix, create of RSM surface matrix, analyze factorial design and analyze response surface design RSM) were performed using Minitap version 17 software. All results of starch yield and bioplastic property (opacity, solubility and biodegradability) were calculated using Microsoft Excel. One-way ANOVA analyses of variance were used to compare the optimal bioplastic, standard bioplastic and commercial plastic and multiple comparison of means by Tukey test were performed using SPSS version 27. The significant level for all analyses was 0.05.

Chapter 2

Results and Discussion

CHAPTER 2: RESULTS AND DISCUSSION

2.1. Starch yield from wheat bran

2.1.1. Appearance of the extracted starch

The extracted starch was beige white powder, The texture was smooth and fine, completely dry and pure, compared to commercial starch, it had a slightly different color, smell, and taste, but it remained stable and easy to store.

Source: *original (2025).*

Figure (11). The extracted starch from wheat bran.

2.1.2. Starch yield results

The starch yield extracted from wheat bran was 23.14±2.85 %. This result is lower compared to reported by Sardari et *al.* (2019). In their research, yields reached 68.2% for fine bran and 81.7% for coarse bran under similar extraction conditions. The differences could be due to several factors. According to Rhazi et *al.* (2021), starch content can vary significantly between wheat cultivars. Also, these differences may be related to genetic factors and climatic conditions, which affect starch synthesis in the plant (Vignola et *al.*, 2016). While our result is superior to that obtained by Babu et *al.* (2014), where the solvent used in the extraction influences the yield. The authors confirmed that using distilled water as a solvent produces more yield.

2.2. General appearance of standard starch-based bioplastic

The starch-based bioplastic produced under standard conditions exhibited a brown color with a slightly transparent appearance. The surface was glossy and smooth, and no visible defects such as cracks or air bubbles. The film texture felt dry, and the thickness was not uniformed due to the parchment paper. The bioplastic produced was bendable and elastic. Moreover, the films remained flat and stable over days.

Source: *original (2025).*

Figure (12): General appearance of standard starch-based bioplastic.

2.3. Evaluation and comparison of the performance of starch-based bioplastics and commercial plastic

2.3.1. Opacity

The standard starch-based bioplastic showed moderate transparency (5.31), making it suitable for semi-transparent packaging. The opacity of 12 bioplastics produced according to Plackett-Burman matrix indicated variable results between 2.55 and 7.03. While in commercial plastic the opacity is high compared to bioplastics produced, was 16.00 (Table 05).

When looking for good transparency to compare between plastics. Among the 12 films developed according to the Plackett-Burman matrix, The biofilm with the lowest opacity (B12, 2.55%) was selected as the most optimal and compared to the standard bioplastic and commercial plastic.

ANOVA confirmed that there were significant differences between the three film types (F = 4129.03, $P < 0.001$), and multiple comparison of means with Tukey's test identified statistically distinct groups (Figure 13).

Table (05). Opacity test results.

Cuvette content	Opacity (%)
Commercial plastic	16.00
Standard	5.31
B1	2.71
B2	7.03
B3	3.76
B4	3.93
B5	5.60
B6	4.20
B7	3.42
B8	2.75
B9	2.80
B10	3.78
B11	3.65
B12	2.55

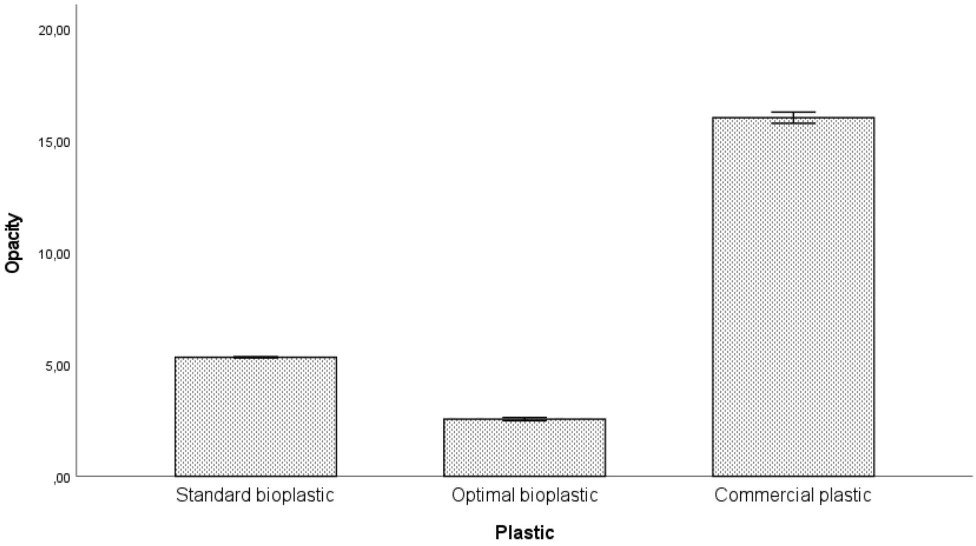


Figure (13). Opacity of commercial plastic, standard bioplastic, and starch-based bioplastics (B1-B12).

The commercial plastic showed the highest opacity (16.00), indicating its strong light-blocking properties (Shah et *al.*, 2008). While the standard starch-based bioplastic showed a moderate opacity (5.31), and the optimal film demonstrated the lowest opacity (2.55), suggesting better transparency. Despite these two films were produced by the same method, but the difference in the amount of citric acid and the drying process makes a significant difference between them. Therefore, the improved transparency in the optimal film can be attributed to both the higher citric acid content and the milder drying process. According Melro et *al.* (2020), Citric acid, when used in greater amounts, may have enhanced the compatibility between film components, improving polymer dispersion and resulting in a smoother and more homogeneous surface that allows lighter to pass through.Thus, a bioplastic with lower opacity is considered better, as it indicates improved transparency and a more uniform structure

2.3.2. Solubility

Water solubility values of the bioplastic produced by Plackett Burman Matrix (Table 06) ranged from 28.85 % (B1) to 50.77 % (B9). The standard film exhibited a solubility of 30.00 % and commercial plastic recorder no water solubility 0%, indicating perfect water resistance.

Table (06). Water solubility test results.

Biofilm type	**solubility (%)**
Commercial plastic	0.00
Standard	30.00
B1	28.85
B2	39.13
B3	42.11
B4	36.11
B5	42.86
B6	42.55
B7	35.42
B8	37.50
B9	50.77
B10	33.33
B11	31.82
B12	49.06

ANOVA confirmed that there were significant differences between the three film types (F = 5861.13, P < 0.001), and multiple comparison of means with Tukey's test identified statistically distinct groups (Figure 14).

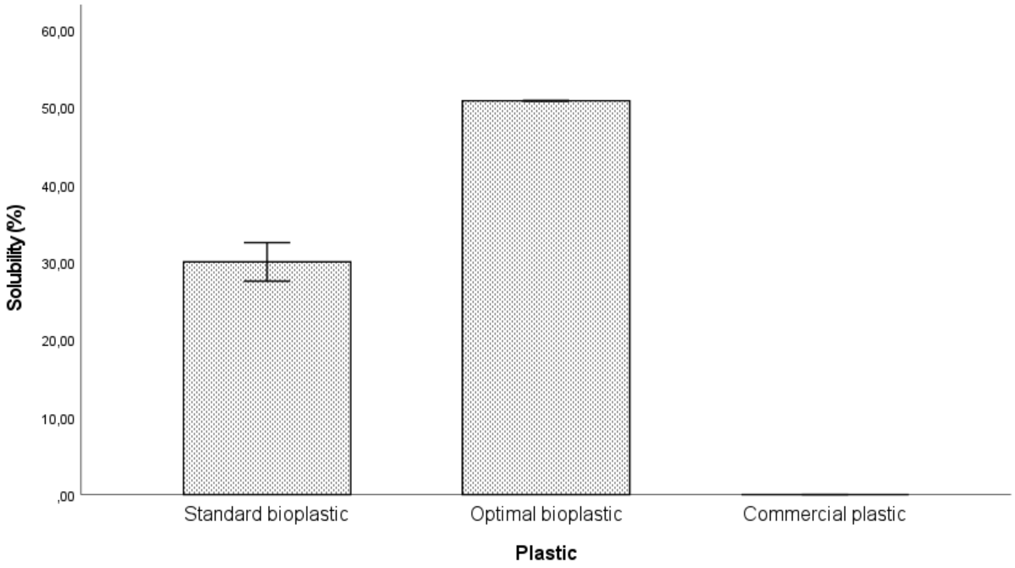


Figure (14). Water solubility percentages of commercial plastic, standard bioplastic, and optimal bioplastics (B9).

As expected, the commercial plastic exhibited zero solubility, confirming its complete resistance to water. The solubility results revealed significant differences between the standard bioplastic and the most soluble bioplastic. The latter, although composed of the same base materials, contained a lower amount of citric acid (0.5 g) and was dried at a lower temperature (70 °C) for a longer period (3 hours).

The increased solubility in this optimal film can be explained by several factors. First, citric acid can act as a cross-linking agent, forming ester bonds with hydroxyl groups in starch, which reduces the film's water affinity (Reddy & Yang, 2010). However, with only 0.5 g of citric acid, the degree of cross-linking was likely insufficient, resulting in a more hydrophilic structure that allowed easier water penetration and dissolution.

Second, drying at a lower temperature may have led to incomplete starch gelatinization and higher residual moisture content, both of which are known to increase water sensitivity (González-Seligra et *al.*, 2016).

While high solubility, as observed in the optimal film, could be advantageous for biodegradable packaging requiring rapid disintegration, it would be less appropriate for high-moisture or long-shelf-life applications.

2.3.3. Biodegradability

Results of biodegradability varied significantly across the samples, from 3.70% (Standard) and 75.00% (more degradable), while the commercial plastic remained undegraded (0%).

Table (07). Biodegradability test results.

Biofilm type	Biodegradability (%)
Commercial plastic	0.00
Standard	3.70
B1	32.14
B2	54.55
B3	74.19
B4	45.95
B5	52.94
B6	75.00
B7	42.86
B8	68.00
B9	36.59
B10	30.30
B11	20.00
B12	8.33

ANOVA confirmed that there were significant differences between the three film types (F = 6351.44, P < 0.001), and multiple comparison of means with Tukey's test identified statistically distinct groups (Figure 15).

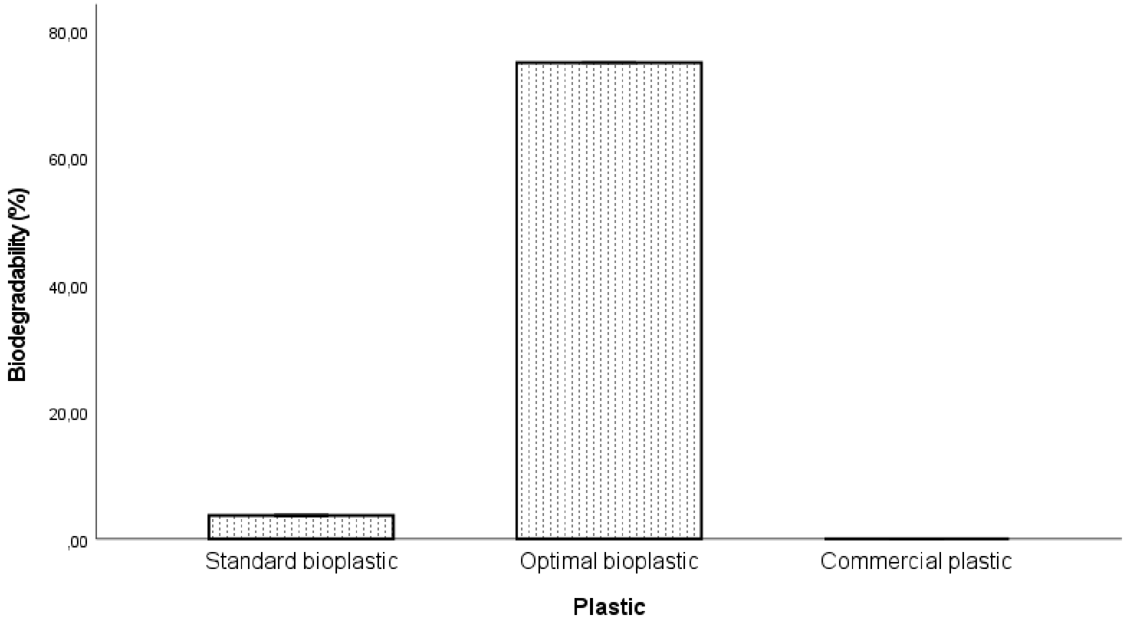


Figure (15). Biodegradability percentages of commercial plastic, standard bioplastic, and optimal bioplastics (B6).

The significant difference in biodegradability between the standard and optimal bioplastic films can be attributed to both formulation differences and drying conditions. The optimal film was prepared with the same amounts of starch and plasticizer using the standard protocol, but with a high amount of citric acid. It was also dried at a higher temperature of 110 °C and for a long time of 3 hours. The combined effect of higher temperature and extended drying time, which may have enhanced starch gelatinization and reduced residual moisture, leading to a more fragile and porous film structure that microbes can break down more easily (González-Seligra et *al.*, 2016). The increased citric acid may also have contributed to better plasticization and structural openness, facilitating microbial access (Souza et *al.*, 2012). The commercial plastic, as expected, didn’t degrade at all because it’s made of synthetic, non-biodegradable material.

2.4. Results of analyse factorial design of biodegradability

For all the variables tested, the analysis factorial design showed that drying temperature and drying time were the factors that had significant positive effects on biodegradability ($P<0,05$) (Table 08). Whereas, other factors namely plasticizer type and amount of citric acid showed negative effects on biodegradability.

Table (08). Estimates of the model coefficients.

Model	**Coefficient**	**T- value**	**P- value**	**Signification**
Constant	47.49	15.45	0.000	**Significant**
Plasticizer type	1.33	0.43	0.678	Non-significant
Quantity of citric acid	-0.30	-0.10	0.925	Non-significant
Drying temperature	**9.42**	**3.06**	**0.018**	**Significant**
Drying time	**10.76**	**3.50**	**0.010**	**Significant**

The Pareto diagram (figure 16) shows the most important factors among all the factors studied at the chosen confidence level ($\alpha = 0.05$), and ranks them in order of importance. When the reference line P (2.571) intersects the bars, this indicates that it separates the factors with significant effects at the threshold of $P < 0.05$ from those with non-significant effects. The lengths of the bars are proportional to the importance of the effects of the factors. Drying time is therefore the most significant factor followed by drying temperature with a positive effect.

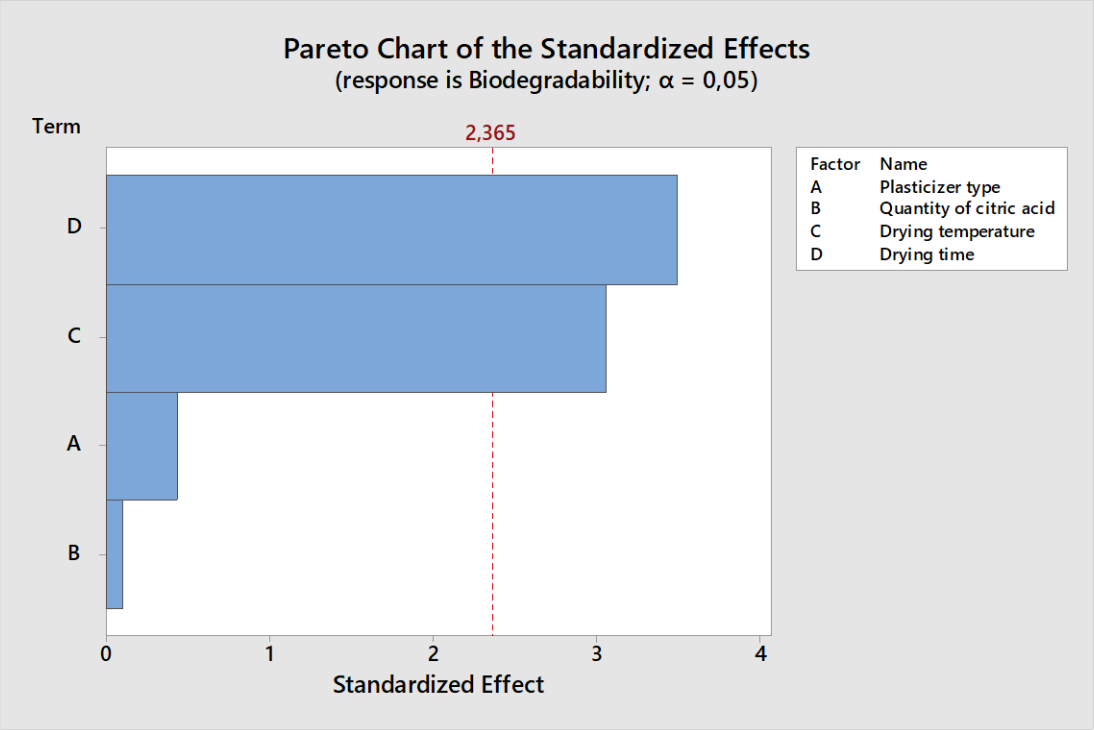


Figure (16): Pareto chart for biodegradability.

The regression equation confirms this trend:

Biodegradability = 47.49 + 1.33 (Plasticizer) – 0.30 (Citric Acid) + 9.42 (Temperature) + 10.76 (Time).

The factorial analysis revealed that drying time/temperature were the only factors with statistically significant positive effects on the biodegradability of starch-based bioplastics. Drying time emerged as the most influential variable according to the Pareto chart, followed closely by drying temperature. The increased exposure to heat over a longer period likely promotes more extensive gelatinization of starch and reduces residual moisture. These structural changes contribute to the formation of a more porous and brittle film, which facilitates microbial penetration and enzymatic activity, as supported by González-Seligra et *al.* (2016) and Souza et *al.* (2012).

2.5. Optimization Using Central Composite Design (RSM)

The experimental design included 13 runs with coded values for each variable (−1, 0, +1, ±1.414), which were converted to real values (°C and hours). These runs were used to model the biodegradability behavior of starch-based bioplastics under different processing conditions.

Table (09). Central Composite Design Matrix: Coded and Actual Values of Drying Temperature and Time with Experimental Biodegradability

Run Order	Coded Temp	Coded Time	Experimental Biodegradability (%)
1	0	0	45.95
2	0	0	42.22
3	-1	1	43.59
4	1.414	0	55.17
5	0	-1.414	35.42
6	1	1	55.17
7	-1.414	0	20.69
8	-1	-1	26.09
9	0	1.414	60.47
10	0	0	31.82
11	0	0	45.65
12	1	-1	36.36
13	0	0	51.16

According the table 10, the analysis response surface design showed that there were no significant interactions between the factors on biodegradability.

Table (10). Analysis of Variance (RSM).

Term	Coefficient	T- value	P- value
Constant	43,36	2,98	0,000
Drying temperature	8,83	2,36	0,007
Drying time	8,97	2,53	0,007
Drying temperature*Drying temperature	3,37	2,53	0,224
Drying time*Drying time	1,63	3,34	0,539
Drying temperature*Drying time	0,33	3,34	0,925

The significant variables were applied to the RSM-CCD approach. Regression was achieved to adequate the response function to the experimental values and has led to the model represented by the following Equation:

Biodegradability (%) = $43.36 + (6.24 \times \text{Temperature}) + (6.34 \times \text{Time}) - (1.69 \times \text{Temperature}^2) + (0.82 \times \text{Time}^2) + (0.16 \times \text{Temperature} \times \text{Time})$

Model Performance Analysis

The 3D response surface plots and contour plots are presented in Figures (17) and (18). The correlation between drying time and drying temperature, and the evaluation of their optimal values are defined both by the 3D graphical representations and the response surface diagram. Interactions between the two important variables were shown in Figure (17). The plot showing the levels of drying time versus drying temperature indicated that the biodegradfability of bioplastic increased along the elevation of drying teme and temprature levels (Figure 17).

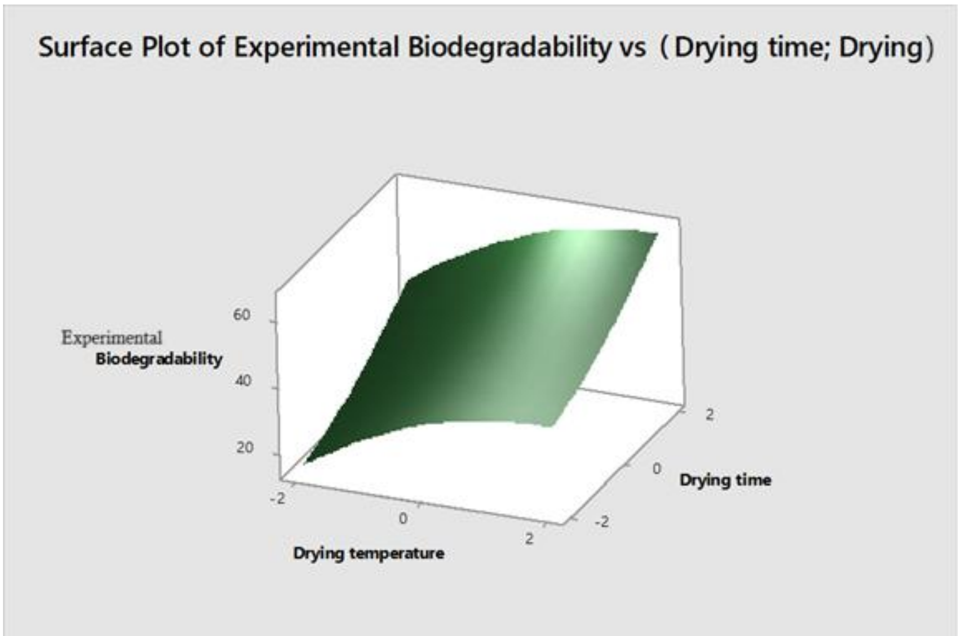


Figure (17). Response surface plots of the effects of various factors (Drying time and drying temperature) on the biodegradability of bioplastic.

In Figure (18), contour plots showed circular presentation; in consequence, the interaction between drying time and drying temperature has not a significant effect in the biodegradability in the soil. The optimum conditions for a the biodegrdability were defined through the interpretation of the response surface plots in addition to the regression equation (1).

Concentric circular contour plots were obtained (Figure 18). This indicated that the interaction between drying time and drying temperature has a significant positive impact on the biodegradability process. Nevertheless, the maximum of biodegradability reached the highest level of 60.47% Table (09).

The coefficient of determination R^2 = 81,53% was sufficient and gave acceptable compatibility with the experimental values from the proposed mathematical model. However, the adjusted R^2 (68.34%) and predicted R^2 (36.82%) suggest that the model's predictive power remains moderate and could be improved with additional influencing factors.

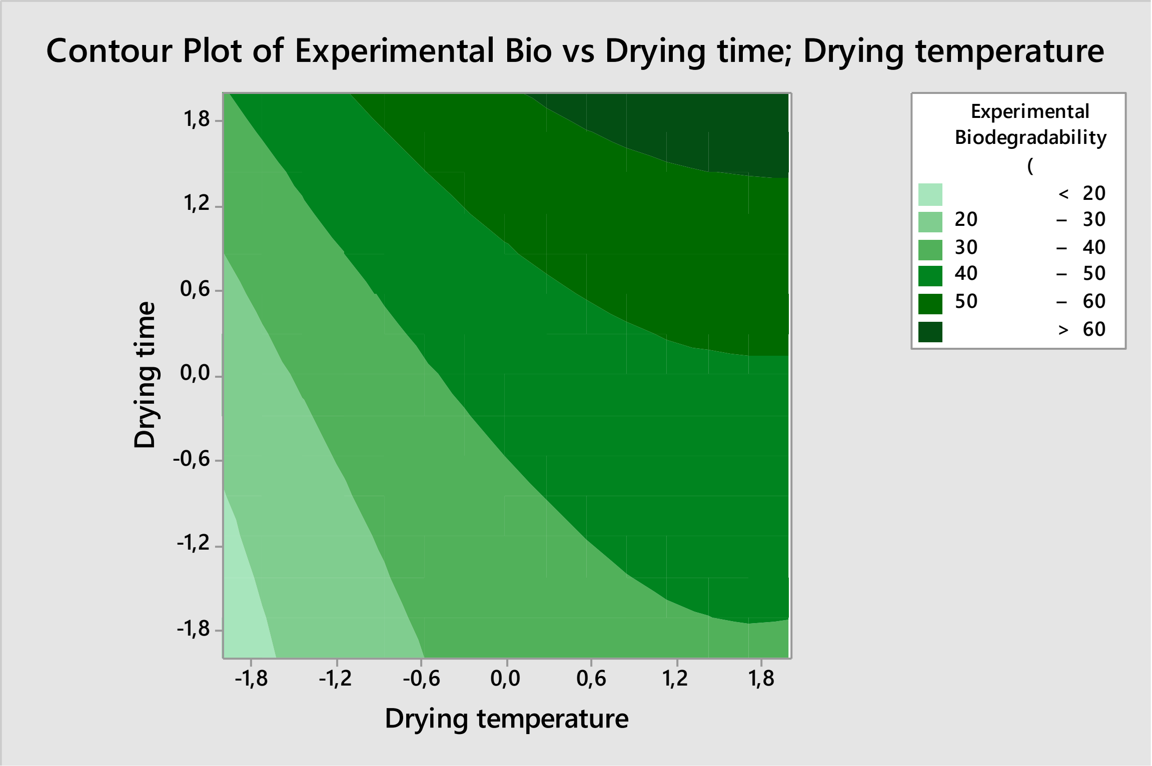


Figure (18). Contour Plot of the biodegradability of bioplastic.

Conclusion

Conclusion

At the end of this research, titled "Optimization of a Production Process for Food Bio-Packaging Based on Cereal By-Products," several important results were obtained regarding starch extraction and its use as a sustainable raw material for the production of bioplastics. In addition, the development of optimized bioplastic film, biodegradable and of good physical quality through systematic experimental design methods, including Plackett-Burman screening and response surface methodology (RSM).

The successful extraction of starch from wheat bran, with a yield of 23.14 ± 2.85%, confirmed its viability as a renewable feedstock. But this can be affected and varied by several factors.

Furthermore, optimized films, consisting of a good combination of factors, demonstrated improved transparency, acceptable water solubility, and microbial biodegradability, indicating their suitability for eco-friendly packaging applications, particularly where environmental performance is a priority. For example, these films could be used for single-use food packaging such as fruit and vegetable wraps, dry snack pouches, bakery bags, or compostable liners. Conversely, commercial plastic was distinguished by a marked total resistance to biodegradability in water and compost.

The study revealed that drying temperature and drying time are the most influential factors affecting film biodegradability. Higher temperatures and extended drying durations significantly enhanced the films' susceptibility to microbial degradation, resulting in biodegradability rates as high as 75%. The RSM model achieved a coefficient of determination (R^2) of 81.53%, indicating a good fit with the experimental data. However, the adjusted R^2 (68.34%) and predicted R^2 (36.82%) suggest that the model's predictive power remains moderate and could be improved with additional influencing factors.

In perspective, some points to complete the study were added:

- Further studies are necessary to evaluate other important properties such as mechanical strength, thermal stability, and barrier behavior in order to assess the material's suitability for practical applications.
- This method can also be applied to other agricultural wastes, such as rice husks, corn stalks, sugarcane bagasse, potato peels, and fruit pomace, to support recycling in agriculture and create more types of eco-friendly bioplastics from different plants.

References

1. Asfaw, W. A., Tafa, K. D., & Satheesh, N. (2023). Optimization of citron peel pectin and glycerol concentration in the production of edible film using response surface methodology. Heliyon, 9(3), e13724. https://doi.org/10.1016/j.heliyon.2023.e13724

2. Ashok, A., & Rejeesh, C. R. (2019). Investigating the Biodegradability and Physical Properties of Starch Derived Bioplastic Films Reinforced with Nanosilica. International Journal of Nanoscience, 18(06), 1850037. https://doi.org/10.1142/S0219581X18500370

3. Babu, A. S., Parimalavalli, R., Jagannadham, K., & Rao, J. S. (2015). Chemical and structural properties of sweet potato starch treated with organic and inorganic acid. Journal of Food Science and Technology, 52(9), 5745–5753. https://doi.org/10.1007/s13197-014-1650-x

4. Organisation for Economic Co-operation and Development. (2014). Biobased chemicals and bioplastics: Finding the right policy balance (OECD Science, Technology and Industry Policy Papers No. 17). https://doi.org/10.1787/5jxwwfjx0djf-en

5. Casas, G. A., Rodriguez, D. A., & Stein, H. H. (2018). Nutrient composition and digestibility of energy and nutrients in wheat middlings and red dog fed to growing pigs1. Journal of Animal Science, 96(1), 215–224. https://doi.org/10.1093/jas/skx01

6. Danciu, C.-A., Tulbure, A., Stanciu, M.-A., Antonie, I., Capatana, C., Zerbeș, M. V., Giurea, R., & Rada, E. C. (2023). Overview of the Sustainable Valorization of Using Waste and By-Products in Grain Processing. Foods, 12(20), 3770. https://doi.org/10.3390/foods12203770

7. Dziki, D. (2023). The Latest Innovations in Wheat Flour Milling: A Review. Agricultural Engineering, 27(1), 147–162. https://doi.org/10.2478/agriceng-2023-0011

8. EUBIO. (2023). Bioplastics. European Bioplastics e, from https://www.european-bioplastics.org/bioplastics/

9. García, Á. (2025, April 15). Nutritional characteristics of wheat bran as animal feed | Dellait. Dellait. https://dellait.com/nutritional-characteristics-of-wheat-bran-as-animal-feed/,

10. Hemery, Y., Rouau, X., Lullien-Pellerin, V., Barron, C., & Abecassis, J. (2007). Dry processes to develop wheat fractions and products with enhanced nutritional quality. Journal of Cereal Science, 46(3), 327–347. https://doi.org/10.1016/j.jcs.2007.09.008

11. Hossain, K., Ulven, C., Glover, K., Ghavami, F., Simsek, S., Alamri, M. S., Kumar, A., & Mergoum, M. (2018). Interdependence of Cultivar and Environment on Fiber Composition in Wheat Bran.

12. Huang, Q., Piao, X. S., Ren, P., & Li, D. F. (2012). Prediction of Digestible and Metabolizable Energy Content and Standardized Ileal Amino Acid Digestibility in Wheat Shorts and Red Dog for Growing Pigs. Asian-Australasian Journal of Animal Sciences, 25(12), 1748–1758. https://doi.org/10.5713/ajas.2012.12298

13. Kumar, P., Yadava, R. K., Gollen, B., Kumar, S., Verma, R. K., & Yadav, S. (n.d.). Nutritional Contents and Medicinal Properties of Wheat: A Review. 2011.

14. Marichelvam, M. K., Jawaid, M., & Asim, M. (2019). Corn and Rice Starch-Based Bio-Plastics as Alternative Packaging Materials. Fibers, 7(4), 32. https://doi.org/10.3390/fib7040032

15. Mateo Anson, N., Hemery, Y. M., Bast, A., & Haenen, G. R. M. M. (2012). Optimizing the bioactive potential of wheat bran by processing. Food & Function, 3(4), 362–375. https://doi.org/10.1039/c2fo10241b

16. Melro, E., Antunes, F. E., Da Silva, G. J., Cruz, I., Ramos, P. E., Carvalho, F., & Alves, L. (2020). Chitosan Films in Food Applications. Tuning Film Properties by Changing Acidic Dissolution Conditions. Polymers, 13(1), 1. https://doi.org/10.3390/polym13010001

17. Mohammed, A. A. B. A., Omran, A. A. B., Hasan, Z., Ilyas, R. A., & Sapuan, S. M. (2021). Wheat Biocomposite Extraction, Structure, Properties and Characterization: A Review. Polymers, 13(21), 3624. https://doi.org/10.3390/polym13213624

18. Myers, R. H., Montgomery, D. C., & Anderson-Cook, C. M. (2016). Response surface methodology: Process and product optimization using designed experiments (4th ed.). John Wiley & Sons.

19. Oluwasina, O. O., Olaleye, F. K., Olusegun, S. J., Oluwasina, O. O., & Mohallem, N. D. S.

(2019). Influence of oxidized starch on physicomechanical, thermal properties, and atomic force micrographs of cassava starch bioplastic film. International Journal of Biological Macromolecules, 135, 282–293. https://doi.org/10.1016/j.ijbiomac.2019.05.150

20. Onipe, O. O., Jideani, A. I. O., & Beswa, D. (2015). Composition and functionality of wheat bran and its application in some cereal food products. International Journal of Food Science & Technology, 50(12), 2509–2518. https://doi.org/10.1111/ijfs.12935

21. Pathak, S., Sneha, C., & Mathew, B. B. (n.d.). Bioplastics: Its Timeline Based Scenario & Challenges.

22. Prückler, M., Siebenhandl-Ehn, S., Apprich, S., Höltinger, S., Haas, C., Schmid, E., & Kneifel, W. (2014). Wheat bran-based biorefinery 1: Composition of wheat bran and strategies of functionalization. LWT - Food Science and Technology, 56(2), 211–221. https://doi.org/10.1016/j.lwt.2013.12.004

23. Raghu Babu, C., Ketanapalli, H., Beebi, S. K., & Kolluru, V. C. (2018). Wheat Bran-Composition and Nutritional Quality: A Review. Advances in Biotechnology & Microbiology, 09(1). https://doi.org/10.19080/AIBM.2018.09.555754

24. Reddy, N., & Yang, Y. (2010). Citric acid cross-linking of starch films. Food Chemistry, 118(3), 702–711. https://doi.org/10.1016/j.foodchem.2009.05.050

25. Reynolds, M. P., & Braun, H.-J. (2022). Wheat Improvement. In M. P. Reynolds & H.-J. Braun (Eds.), Wheat Improvement (pp. 3–15). Springer International Publishing. https://doi.org/10.1007/978-3-030-90673-3_1

26. Rhazi, L., Méléard, B., Daaloul, O., Grignon, G., Branlard, G., & Aussenac, T. (2021). Genetic and Environmental Variation in Starch Content, Starch Granule Distribution and Starch Polymer Molecular Characteristics of French Bread Wheat. Foods, 10(2), 205. https://doi.org/10.3390/foods10020205

27. Rhim, J.-W., Lee, J.-H., & Kwak, H.-S. (2005). Mechanical and water barrier properties of soy protein and clay mineral composite films. Food Science and Biotechnology, 14(1), 112–116.

28. Rosa, N. N., Barron, C., Gaiani, C., Dufour, C., & Micard, V. (2013). Ultra-fine grinding

increases the antioxidant capacity of wheat bran. Journal of Cereal Science, 57(1), 84–90. https://doi.org/10.1016/j.jcs.2012.10.002

29. Rosenboom, J.-G., Langer, R., & Traverso, G. (2022). Bioplastics for a circular economy. Nature Reviews Materials, 7(2), 117–137. https://doi.org/10.1038/s41578-021-00407-8

30. Rossi, D., Rossi, S., Cinelli, P., & Seggiani, M. (2024). Emerging opportunities in the valorisation of wheat bran byproduct as additive in polymer composite materials. Materials Today Sustainability, 27, 100832. https://doi.org/10.1016/j.mtsust.2024.100832

31. Saini, J. K., Saini, R., & Tewari, L. (2015). Lignocellulosic agriculture wastes as biomass feedstocks for second-generation bioethanol production: Concepts and recent developments. 3 Biotech, 5(4), 337–353. https://doi.org/10.1007/s13205-014-0246-5

32. Sanyang, M. L., Ilyas, R. A., Sapuan, S. M., & Jumaidin, R. (2018). Sugar Palm Starch-Based Composites for Packaging Applications. In M. Jawaid & S. K. Swain (Eds.), Bionanocomposites for Packaging Applications (pp. 125–147). Springer International Publishing. https://doi.org/10.1007/978-3-319-67319-6_7

33. Sardari, R. R. R., Sutiono, S., Azeem, H. A., Galbe, M., Larsson, M., Turner, C., & Nordberg Karlsson, E. (2019). Evaluation of Sequential Processing for the Extraction of Starch, Lipids, and Proteins from Wheat Bran. Frontiers in Bioengineering and Biotechnology, 7, 413. https://doi.org/10.3389/fbioe.2019.00413

34. Senapati, N., Semenov, M. A., Halford, N. G., Hawkesford, M. J., Asseng, S., Cooper, M., Ewert, F., Van Ittersum, M. K., Martre, P., Olesen, J. E., Reynolds, M., Rötter, R. P., & Webber, H. (2022). Global wheat production could benefit from closing the genetic yield gap. Nature Food, 3(7), 532–541. https://doi.org/10.1038/s43016-022-00540-9

35. Shah, A. A., Hasan, F., Hameed, A., & Ahmed, S. (2008). Biological degradation of plastics: A comprehensive review. Biotechnology Advances, 26(3), 246–265. https://doi.org/10.1016/j.biotechadv.2007.12.005

36. Shewry, P. R., & Halford, N. G. (2002). Cereal seed storage proteins: Structures, properties and role in grain utilization. Journal of Experimental Botany, 53(370), 947–958. https://doi.org/10.1093/jexbot/53.370.947

37. Shewry, P. R., & Hey, S. J. (2015). The contribution of wheat to human diet and health. Food and Energy Security, 4(3), 178–202. https://doi.org/10.1002/fes3.64

38. Sidek, I. S., Draman, S. F. S., Abdullah, S. R. S., & Anuar, N. (2019). Current development on bioplastics and its future prospects: an introductory review. INWASCON Technology Magazine, 03–08. https://doi.org/10.26480/itechmag.01.2019.03.08

39. Siraj, N. (2022). Wheat germ oil: A comprehensive review. Food Science and Technology, 42, e113721. https://doi.org/10.1590/fst.113721

40. Stuart, P. R., & El-Halwagi, M. M. (2012). Integrated biorefineries: Design, analysis, and optimization. CRC press.

41. Sztupecki, W., Rhazi, L., Depeint, F., & Aussenac, T. (2023). Functional and Nutritional Characteristics of Natural or Modified Wheat Bran Non-Starch Polysaccharides: A Literature Review. Foods, 12(14), 2693. https://doi.org/10.3390/foods12142693

42. The State of Food Security and Nutrition in the World 2023. (2023). FAO; IFAD; UNICEF; WFP; WHO; https://doi.org/10.4060/cc3017en

43. United Nations Environment Programme. (2021). From pollution to solution: A global assessment of marine litter and plastic pollution. Nairobi: UNEP. https://www.unep.org/resources/pollution-solution-global-assessment-marine-litter-and-plastic-pollution

44. Wheat Production by Country 2025. (2025). Retrieved 26 June 2025, from https://worldpopulationreview.com/country-rankings/wheat-production-by-country

45. Zhang, A. R., Wei, M., Yan, L., Zhou, G. L., Li, Y., Wang, H. M., Yang, Y. Y., Yin, W., Guo, J. Q., Cai, X. H., Li, J. X., Zhou, H., & Liang, Y. X. (2022). Effects of feeding solid-state fermented wheat bran on growth performance and nutrient digestibility in broiler chickens. Poultry Science, 101(1), 101402. https://doi.org/10.1016/j.psj.2021.101402

46. Zhao, Y., Zhao, C., Tang, X., Zhou, J., Li, H., Zhang, H., & Liu, J. (2021). Physicochemical properties and microstructure of corn flour–cellulose fiber extrudates. Food Science & Nutrition, 9(5), 249.