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Influence of Drying Pretreatment on Oven and Solar Drying of Cassava Tubers (*Manihot esculenta*)

DISSERTATION THESIS

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Declaration

I hereby declare that I have completed this thesis entitled "**Influence of Drying Pretreatment on Oven and Solar Drying of Cassava Tubers (*Manihot esculenta*)**" independently, except for the jointly authored publications that are included. In the case of such publications, my specific contributions have been clearly stated at the start of the relevant publication chapter. Furthermore, I confirm that proper acknowledgement has been provided within this thesis for any references made to the works of others, I also ensure that this work has not been, nor is it currently submitted, for any other degree, to this or any other university. All information sources have been quoted and acknowledged by means of complete references.

In Prague September 1st, 2025

Ellyas Alga Nainggolan

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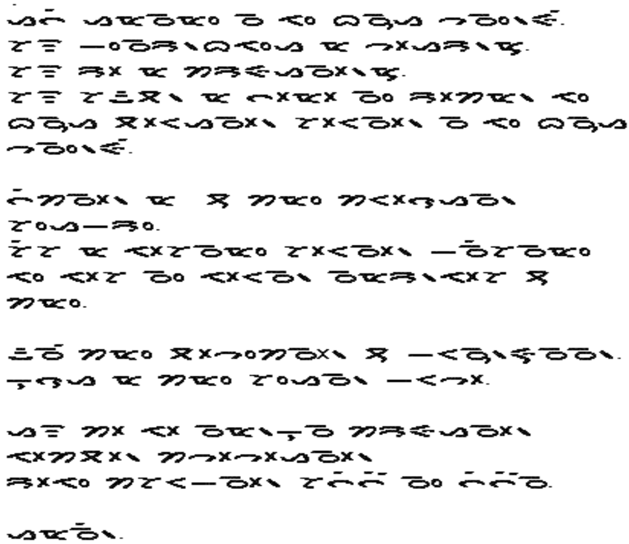

Abstract

This study presents an integrated approach to optimizing the production of cassava flour through a comprehensive evaluation of pretreatment methods and drying systems. Fresh cassava tubers were first subjected to various pretreatments—including chemical treatments using citric acid and sodium metabisulfite, thermal blanching, and water soaking—to modify their physicochemical properties prior to drying. The primary objectives were to: (1) evaluate the effects of different pretreatment methods in combination with two drying systems (solar and oven drying) on the quality of cassava flour; (2) optimize pretreatment and drying parameters specifically for the oven drying system using response surface methodology (RSM) and central composite design (CCD); (3) identify suitable mathematical models to accurately describe the drying kinetics of pretreated cassava slices; and (4) assess the impact of the optimized pretreatment parameters when applied to the solar drying system.

In the oven drying experiments, cassava slices were dried at temperatures ranging from 45.85 to 74.14 °C and for durations between 3.96 and 11.03 hours. Multiple pretreatment configurations also were implemented, notably a chemical-thermal treatment that combined soaking in a solution of 1.31% citric acid and 1.03% sodium metabisulfite followed by a brief blanching period of approximately 1.01 minutes, and a water soaking treatment. Optimization via RSM revealed that the optimal oven drying conditions were achieved at 70 °C for 10 hours, resulting in a cassava flour with a significantly reduced moisture content and an enhanced whiteness index. These quality enhancements were evidenced by reduced hydrogen cyanide content, improved mineral retention, and enhanced

sensory attributes, particularly texture and taste, when the optimized pretreatment was also applied to solar drying system.

Drying kinetics were rigorously modeled using several established thin layers drying models, including the Henderson and Pabis, Page, Wang and Singh, Newton, and logarithmic models. Among these, the logarithmic model provided the best fit for oven drying system, exhibiting the highest coefficient of determination and the lowest error metrics. This finding suggests that the logarithmic model is particularly effective in describing moisture removal from pretreated cassava slices under the optimized conditions. Subsequently, the optimized pretreatment parameters determined from the oven drying trials were applied to a solar drying system. In solar drying experiments, the same pretreatment strategy significantly enhanced drying kinetics by increasing the effective moisture diffusivity and reducing the activation energy, with the Wang and Singh model providing the best fit to the drying data. Moreover, the solar-dried cassava flour exhibited improved physicochemical properties, including enhanced brightness, superior color retention, and a more favorable microstructure. Scanning electron microscopy and Fourier-transform infrared spectroscopy analyses revealed that the optimized chemical–thermal pretreatment preserved the structural integrity of the starch granules, thereby improving hydration and solubility properties of the flour.

Overall, this study demonstrates that the integration of optimized pretreatment techniques with tailored drying protocols can markedly improve cassava flour quality. The successful application of statistical optimization methods and mathematical modeling not only streamlines the drying process but also supports the development of sustainable and energy-

efficient cassava processing strategies for both industrial and small-scale applications. The combined findings offer a promising pathway for enhancing the nutritional, sensory, and safety attributes of cassava flour, thereby expanding its applicability in food, feed, and industrial sectors.

Keywords: cassava flour, pretreatment, drying kinetics, response surface methodology, physicochemical properties, sensory evaluation.

Abstrakt

Tato studie představuje integrovaný přístup k optimalizaci výroby maniokové mouky prostřednictvím komplexního hodnocení metod předúpravy a sušicích systémů. Čerstvé maniokové hlízy byly nejprve vystaveny různým typům předúpravy, chemické úpravě s použitím kyseliny citronové a disiřičitanu sodného, tepelnému blanšírování a namáčení ve vodě, s cílem upravit jejich fyzikálně-chemické vlastnosti před sušením. Hlavními cíli byly: (1) vyhodnotit účinky různých metod předúpravy v kombinaci se dvěma sušicími systémy (solárním a sušením v sušárně) na kvalitu maniokové mouky; (2) optimalizovat parametry předúpravy a sušení specificky pro systém sušení v sušárně pomocí metody odezvových ploch (RSM) a centrálně kompozitního návrhu (CCD); (3) identifikovat vhodné matematické modely, které přesně popisují kinetiku sušení předupravených plátků manioku; a (4) posoudit vliv optimalizovaných parametrů předúpravy aplikovaných v systému solárního sušení.

Při experimentech se sušením v sušárně byly plátky manioku sušeny při teplotách v rozmezí od 45,85 do 74,14 °C po dobu od 3,96 do 11,03 hodin. Rovněž byly realizovány různé konfigurace předúprav, zejména chemicko-tepelná úprava kombinující namáčení v roztoku 1,31% kyseliny citronové a 1,03% disiřičitanu sodného, následovaná krátkým blanšírováním trvajícím přibližně 1,01 minuty, a úprava namáčením ve vodě. Optimalizace pomocí RSM ukázala, že optimální podmínky sušení v sušárně byly dosaženy při 70 °C po dobu 10 hodin, což vedlo k významně sníženému obsahu vlhkosti maniokové mouky a zlepšenému indexu bělosti. Tyto kvalitativní zlepšení byla potvrzena nižším obsahem kyanovodíku, lepší retencí minerálních látek a vylepšenými

senzorickými vlastnostmi, zejména texturou a chutí, když byla optimalizovaná předúprava aplikována rovněž na systém solárního sušení.

Kinetika sušení byla důkladně modelována pomocí několika zavedených modelů tenké vrstvy, včetně modelů Henderson a Pabis, Page, Wang a Singh, Newton a logaritmického modelu. Z těchto modelů poskytoval logaritmický model nejlepší shodu pro systém sušení v sušárně, vykazující nejvyšší koeficient determinace a nejnižší chyby. Tento výsledek naznačuje, že logaritmický model je zvláště efektivní při popisu odstraňování vlhkosti z předupravených plátků manioku za optimalizovaných podmínek. Optimalizované parametry předúpravy získané z pokusů se sušením v sušárně byly následně aplikovány na systém solárního sušení. Při experimentech se solárním sušením stejná strategie předúpravy významně zlepšila kinetiku sušení zvýšením efektivní difuzivity vlhkosti a snížením aktivační energie, přičemž nejlepší shodu s experimentálními daty poskytoval model Wang a Singh. Solárně sušená manioková mouka navíc vykazovala zlepšené fyzikálně-chemické vlastnosti, včetně lepšího jasu, zachování barvy a příznivější mikrostruktury. Analýzy pomocí rastrovací elektronové mikroskopie (SEM) a Fourierovy infračervené spektroskopie (FTIR) odhalily, že optimalizovaná chemicko-tepelná předúprava zachovává strukturální integritu škrobových granulí, čímž zlepšuje hydrataci a rozpustnost mouky.

Celkově studie dokládá, že integrace optimalizovaných technik předúpravy s cílenými sušicími protokoly může výrazně zvýšit kvalitu maniokové mouky. Úspěšná aplikace metod statistické optimalizace a matematického modelování nejen zefektivňuje proces sušení, ale také podporuje rozvoj udržitelných a

energeticky účinných strategií zpracování manioku pro průmyslové i drobné aplikace. Kombinované výsledky představují slibnou cestu ke zlepšení nutričních, senzorických a bezpečnostních vlastností maniokové mouky, a tím rozšiřují její využitelnost v potravinářském, krmivářském a průmyslovém sektoru.

Klíčová slova: manioková mouka, předúprava, kinetika sušení, metoda odezvových ploch, fyzikálně-chemické vlastnosti, senzorické hodnocení.

Abstrak

Penelitian ini menyajikan pendekatan terpadu untuk mengoptimasi produksi tepung singkong melalui evaluasi komprehensif atas metode pra-perlakuan dan sistem pengeringan. Umbi singkong segar pertama-tama dikenai berbagai pra-perlakuan termasuk perlakuan kimiawi menggunakan asam sitrat dan natrium metabisulfit, pemanasan (blanching), serta perendaman dalam air untuk memodifikasi sifat fisikokimia sebelum proses pengeringan. Tujuan utama penelitian adalah: (1) mengevaluasi pengaruh berbagai metode pra-perlakuan yang dikombinasikan dengan dua sistem pengeringan (pengeringan surya dan oven) terhadap kualitas tepung singkong; (2) mengoptimasi parameter pra-perlakuan dan pengeringan khusus untuk sistem pengeringan oven menggunakan metode permukaan respons (RSM) dan rancangan komposit pusat (CCD); (3) mengidentifikasi model matematika yang sesuai untuk menjelaskan kinetika pengeringan irisan singkong yang telah dipra-perlakukan; dan (4) mengevaluasi dampak parameter pra-perlakuan teroptimasi saat diterapkan pada sistem pengeringan surya.

Dalam percobaan pengeringan oven, irisan singkong dikeringkan pada suhu berkisar antara 45,85 hingga 74,14 °C dan untuk durasi antara 3,96 hingga 11,03 jam. Berbagai konfigurasi pra-perlakuan juga diterapkan, khususnya perlakuan kimia–termal yang menggabungkan perendaman dalam larutan 1,31 % asam sitrat dan 1,03 % natrium metabisulfit diikuti oleh pemanasan cepat (blanching) selama sekitar 1,01 menit, serta perlakuan perendaman air distilasi. Optimasi melalui RSM menunjukkan bahwa kondisi optimal pengeringan oven dicapai pada suhu 70 °C selama 10 jam, menghasilkan tepung singkong dengan kadar air yang secara

signifikan tereduksi dan indeks kecerahan yang meningkat. Peningkatan kualitas tersebut ditandai oleh penurunan kadar hidrogen sianida, peningkatan retensi mineral, serta peningkatan atribut sensorik terutama tekstur dan rasa ketika pra-perlakuan teroptimasi juga diterapkan pada sistem pengeringan surya.

Kinetika pengeringan dimodelkan secara akurat menggunakan beberapa model pengeringan lapis-tipis yang telah mapan, termasuk model Henderson dan Pabis, model Page, model Wang dan Singh, Newton, serta model logaritmik. Di antara model-model tersebut, model logaritmik memberikan kecocokan terbaik untuk sistem pengeringan oven, dengan koefisien determinasi tertinggi dan metrik kesalahan terendah. Temuan ini menunjukkan bahwa model logaritmik sangat efektif dalam menjelaskan mekanisme penghilangan air dari irisan singkong yang telah dipra-perlakukan pada kondisi teroptimasi. Selanjutnya, parameter pra-perlakuan teroptimasi yang diperoleh dari percobaan pengeringan oven diterapkan pada sistem pengeringan surya. Dalam percobaan pengeringan surya, strategi pra-perlakuan yang sama secara signifikan meningkatkan kinetika pengeringan dengan memperbesar difusivitas air efektif dan menurunkan energi aktivasi, dimana model Wang dan Singh memberikan kecocokan terbaik pada data pengeringan. Selain itu, tepung singkong kering surya menunjukkan peningkatan sifat fisikokimia, termasuk peningkatan kecerahan, retensi warna yang lebih baik, serta mikrostruktur yang lebih menguntungkan. Analisis dengan pemindaian mikroskop elektron dan spektroskopi inframerah transformasi fourier mengungkapkan bahwa pra-perlakuan kimia–termal teroptimasi mempertahankan integritas struktural granula pati, sehingga meningkatkan kemampuan hidrasi dan kelarutan tepung.

Secara keseluruhan, penelitian ini membuktikan bahwa integrasi teknik pra-perlakuan teroptimasi dengan protokol pengeringan yang disesuaikan dapat secara nyata meningkatkan kualitas tepung singkong. Penerapan metode optimasi statistik dan pemodelan matematika tidak hanya menyederhanakan proses pengeringan, tetapi juga mendukung pengembangan strategi pengolahan singkong yang berkelanjutan dan hemat energi untuk aplikasi industri maupun skala kecil. Temuan gabungan ini menawarkan jalur menjanjikan untuk meningkatkan atribut nutrisi, sensorik, dan keamanan tepung singkong, sehingga memperluas kegunaannya dalam sektor pangan, pakan, dan industri.

Kata kunci: tepung singkong, pra-perlakuan, kinetika pengeringan, metode permukaan respons, sifat fisikokimia, evaluasi sensorik.

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Abbreviations

a*	Red-Green Axis in Color Measurement
adj R^2	Adjusted Coefficient of Determination
ANOVA	Analysis of Variance
AOAC	Association of Official Analytical Chemists
a_w	Water Activity
b*	Yellow-Blue Axis in Color Measurement
BT	Blanching Time
CA	Citric Acid Concentration
CCD	Central Composite Design
CMFD	Convective Multiple Flash Drying
COP	Coefficient of Performance
CV	Coefficient of Variation
DB1	Chemical-Thermal Pretreatment
D_{eff}	Effective Moisture Diffusivity
DOE	Design of Experiments
E_a	Activation Energy
EHT	Extra High Tension (Used in SEM Analysis)
FC	Fresh Cassava (Control Sample)
FTIR	Fourier-Transform Infrared Spectroscopy
HCN	Hydrogen Cyanide
L*	Brightness in Color Measurement
LPG	Liquefied Petroleum Gas
MC	Moisture Content
MR	Moisture Ratio
OFAT	One-Factor-at-a-Time
PPD	Postharvest Physiological Deterioration
R^2	Coefficient of Determination
RMSE	Root Mean Square Error
RSM	Response Surface Methodology
S3D	Water Soaking Pretreatment
SAHPD	Solar-Assisted Heat Pump Drying

SD	Solar Drying
SEM	Scanning Electron Microscopy
SM	Sodium Metabisulfite Concentration
SMER	Specific Moisture Extraction Ratio
SSE	Sum of Squares Error
T_1	Drying Temperature
T_2	Drying Time
WD	Working Distance (used in SEM Analysis)
WI	Whiteness Index
χ^2	Chi-Square

1. Introduction

1.1. Background

Indonesia has long been recognized as an agrarian nation with a rich agricultural heritage, where cassava (*Manihot esculenta*) plays a pivotal role in ensuring food security and sustaining rural livelihoods. As of 2023, Indonesia produced approximately 17.21 million tons of cassava, making it one of the world's largest cassava producers [1]. In recent years, the country has witnessed a substantial increase in cassava production, not only to meet domestic demand but also to cater to emerging industrial applications. Cassava tubers, rich in starch and essential nutrients, are processed into a variety of value-added products such as tapioca, modified cassava flour (mocaf), and cassava flour [2,3]. Among these, cassava flour stands out as a preferred commodity due to its longer shelf life and diverse culinary applications. However, the intrinsic high moisture content of fresh cassava, often exceeding 60%, renders it highly perishable, leading to rapid postharvest deterioration and quality losses [4]. Consequently, efficient postharvest processing, particularly drying, is imperative for prolonging shelf life and enhancing product quality.

The processing of cassava into flour involves several critical stages that collectively determine the final product's quality and consumer acceptability [5]. Traditional processing methods, though well-ingrained, are increasingly being challenged by modern techniques aimed at minimizing nutrient loss and enhancing physicochemical properties. Drying, a key preservation method, plays a central role in reducing moisture content and inhibiting enzymatic and microbial degradation. Despite its simplicity, the drying process must be carefully controlled to avoid adverse changes in texture, color, and nutritional quality [6]. As such, research efforts have increasingly focused on refining drying parameters and incorporating pretreatment strategies to safeguard the inherent qualities of cassava flour.

Among the various drying methods, solar drying and oven drying have emerged as two promising techniques, each with distinct advantages and challenges. Solar drying harnesses abundant sunlight to provide an energy-efficient and environmentally friendly option, particularly attractive for rural and resource-limited settings [7,8]. However, the variability in solar radiation and ambient conditions can lead to inconsistent drying kinetics and quality variations. Conversely, oven drying offers a more controlled environment where temperature, air velocity, and drying time can be precisely managed, leading to enhanced product uniformity [9]. Nonetheless, oven drying generally involves higher energy consumption and operational costs. The selection of an appropriate drying system thus requires a balanced consideration of energy efficiency, product quality, and economic feasibility [10]. Recent studies have attempted to model the drying kinetics under these different systems to better predict moisture removal rates and optimize processing conditions [11-13].

An equally critical aspect of improving cassava flour quality lies in the effective application of pretreatment methods prior to drying. Pretreatments such as blanching and the use of chemical agents (e.g., citric acid, sodium metabisulfite) have been shown to influence not only the drying behavior but also the structural, physicochemical, and sensory characteristics of the final product [14]. Blanching, by inactivating deleterious enzymes and partially gelatinizing starch, can enhance the rehydration properties and textural attributes of cassava flour

[15]. Chemical pretreatments, meanwhile, are employed to curb enzymatic browning and maintain color stability, though they may also lead to modifications in nutrient composition [16,17]. Additionally, water soaking can reduce harmful constituents but may also result in the leaching of beneficial nutrients. Collectively, these pretreatment methods have a profound impact on drying kinetics by altering the microstructure of cassava tissues, thereby influencing moisture diffusivity and activation energy during the drying process [5].

The optimization of drying parameters is essential to maximizing product quality while minimizing energy consumption. Recent advances in experimental design have facilitated a systematic exploration of the interplay between drying temperature, time, and pretreatment conditions [18,19]. Such approaches allow for the identification of optimal conditions that yield cassava flour with low moisture content and desirable whiteness, as well as favorable textural and nutritional attributes [14]. Mathematical models developed through these studies have proven instrumental in describing the drying kinetics of pretreated cassava slices. Models such as the Page model, Wang and Singh model, and logarithmic models have been used to capture the dynamics of moisture removal accurately, thereby providing insight into the underlying physical processes and serving as valuable tools for scaling up laboratory findings to industrial settings [20-22].

Despite the progress achieved in understanding the individual effects of pretreatment and drying systems, there remains a notable gap in the literature regarding an integrated evaluation of these processes. While previous studies have separately explored the impacts of chemical and thermal pretreatments on cassava drying kinetics and flour quality, comprehensive investigations that combine various pretreatment methods with both solar and oven drying systems are limited. Moreover, there is a need to optimize the drying parameters specifically for the oven drying system using robust experimental designs and subsequently assess whether these optimized pretreatment parameters can be effectively translated to solar drying. Such an integrative approach is crucial for developing a more flexible and sustainable processing protocol that can be adapted to different operational environments and resource availabilities.

1.2. Objectives

1.2.1. Main Objective

The primary objective of this study is to bridge existing research gaps by assessing the effects of chemical and thermal pretreatments, along with solar and oven drying systems, on the physicochemical properties, drying kinetics, and sensory attributes of cassava flour.

1.2.2. Specific Objectives

This study specifically aims to: (1) evaluate the effects of various pretreatment methods, including blanching and chemical agents, as well as different drying systems, such as solar and oven drying, on the quality of cassava flour; (2) optimize pretreatment and drying parameters for the oven drying system; (3) identify suitable mathematical models to describe drying kinetics; and (4) assess the impact of the optimized pretreatment parameters on the solar drying system. By integrating advanced pretreatment strategies with sustainable drying technologies, this research seeks to enhance the nutritional, sensory, and functional properties of cassava flour while promoting environmentally sustainable processing practices.

1.3. Relevance of this Study

Cassava processing represents a critical intersection of food security, sustainability, and industrial innovation. In regions where cassava serves as a dietary staple, improving postharvest processing methods has direct implications for reducing food losses, improving nutrition, and enhancing income generation for smallholder farmers. Moreover, the adoption of sustainable drying technologies, such as solar drying, aligns with global climate goals by reducing energy consumption and minimizing environmental footprints. This study's focus on optimizing pretreatment and drying conditions contributes to the broader discourse on sustainable food processing by proposing practical solutions to the challenges of cassava preservation and value addition.

Cassava's importance as a staple crop necessitates innovative approaches to its postharvest management. By investigating the interplay between pretreatment methods and drying techniques, this study seeks to provide actionable insights for improving cassava flour quality and promoting sustainable processing technologies. The findings will contribute to the development of scalable and energy-efficient cassava processing systems, supporting food security and sustainability efforts in cassava-dependent regions worldwide.

1.4. Structure of the Thesis

This thesis is composed of four main chapters, each representing a detailed scientific study on cassava processing technologies. Below is an overview of each chapter:

Chapter 2: This chapter reviews the recent advancements in the pre-drying, drying, and post-drying processes for cassava tubers. It provides a comprehensive analysis of the existing methodologies and their impacts on the quality and sustainability of cassava products. Challenges and potential future directions in cassava drying techniques are also explored, emphasizing the integration of innovative processes for improved resource utilization and efficiency.

Chapter 3: Focusing on the optimization of drying parameters, this chapter employs the central composite design and superimposition approach to assess the impact of drying temperature and time on pretreated cassava flour. The study highlights the effects of soaking and blanching pretreatments on cassava flour's physicochemical properties, optimizing conditions for sustainable production while preserving product quality.

Chapter 4: This chapter delves into the chemical and thermal pretreatments used in cassava processing, analyzing their effects on moisture content, whiteness index, activation energy, and effective moisture diffusivity. Using advanced modeling and response surface methodology, the study identifies optimal conditions to enhance drying efficiency and cassava flour quality, accompanied by a detailed microstructural analysis.

Chapter 5: The final chapter investigates the role of pretreatment and solar drying on cassava flour. The study examines drying kinetics, thermal properties, and sensory attributes, emphasizing pretreatment methods such as blanching with citric acid and sodium metabisulfite. The findings demonstrate the potential of integrated chemical pretreatments and solar drying in improving cassava flour quality and consumer acceptability, supporting its broader industrial applications.

2. Recent Trends in the Pre-Drying, Drying, and Post-Drying Processes for Cassava Tuber: A Review.

Adapted from: Nainggolan EA, Banout J, Urbanova K (2024) Recent trends in the pre-drying, drying, and post-drying processes for cassava tuber: a review. Foods 13: 1778. https://doi.org/10.3390/foods13111778. IF = 4.7.

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Abstract

Cassava tuber is an essential staple crop in tropical regions with versatile applications in the food, feed, and industrial sectors. However, its high moisture content and perishable nature necessitate efficient preservation methods to extend its shelf life and enhance its value. Pre-drying, drying, and post-drying processes play pivotal roles in maintaining the quality and usability of cassava products. This review comprehensively examines the current status and future directions in the pre-drying, drying, and post-drying processes of cassava tuber. Various pre-drying or pretreatment methods and drying techniques are evaluated for their impacts on drying kinetics and product quality. Additionally, challenges and limitations in achieving high-quality processing of cassava flour are identified. Future directions in cassava drying methods emphasize the integration of combined pre-drying and drying techniques to optimize resource utilization and processing efficiency. Furthermore, the adoption of advanced online measurement and control technologies in drying equipment is highlighted for real-time monitoring and optimization of drying parameters. The importance of optimizing existing processes to establish a comprehensive cassava industrial chain and foster the development of the cassava deep-processing industry is emphasized. This review provides valuable insights into the current trends and future prospects in cassava drying technologies, aiming to facilitate sustainable and efficient utilization of cassava resources for various applications.

Keywords: cassava tuber; pre-drying; drying; post-drying; dried cassava products

2.1. Introduction

Cassava, a staple tuber crop with a rich historical legacy of cultivation, thrives predominantly in tropical regions. In 2021, global cassava production reached an impressive 314 million metric tons [1], with various nations emerging as leaders in its cultivation, as illustrated in Figure 2.1. Currently, cassava plays a crucial role in the agricultural sector of tropical and underdeveloped nations. Parmar et al. [2] reported that cassava is a significant source of calories for over 800 million people in Africa, South America, and Southeast Asia. It plays a vital role in ensuring food security because it can produce approximately 10 tons per hectare, even in unfavorable soil conditions and with few inputs. Cassava tubers provide cost-effective pricing in comparison to other food commodities, and their economic worth can be augmented by means of processing and creating derivative products. Market Data Forecast [3] predicts that the global cassava flour market will reach USD 49.7 billion by 2028, with a compound annual growth rate (CAGR) of 6.4%.

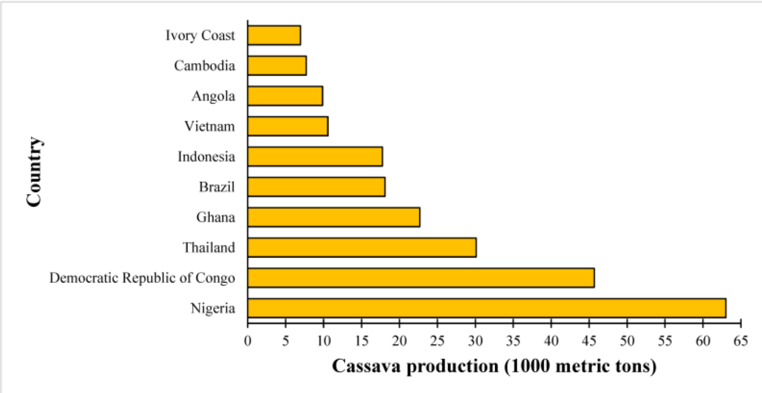

Figure 2.1. Leading cassava-producing nations across the globe in 2021.

Cassava serves not only as a source of carbohydrates but also harbors an array of nutrients such as ascorbic acid, carotenoids, calcium, potassium, iron, magnesium, copper, zinc, and manganese [4]. The presence of carbohydrates in cassava tubers enables their usage in diverse forms and applications [5]. Processing cassava can yield various food items that people can consume directly with simple processing or use as ingredients in food and beverages [6]. In addition, non-food industries make use of cassava, such as in alcohol manufacture, organic acid production, pharmaceuticals, paper manufacturing, and as additives. Traditionally, people often consume cassava tubers by boiling or cooking them, with variations in traditional processing methods observed across different areas and nations [7].

Cassava tubers may display indications of postharvest physiological deterioration (PPD) within 24–72 h of being harvested [8]. Cassava tubers typically have a moisture content of approximately 60–70%, carbohydrates ranging from 30% to 35%, and a protein content of 1% to 2% [9]. The elevated moisture content in cassava tubers impacts the advancement of cassava processing, necessitating prompt processing to generate intermediate products for the purpose of minimizing transportation expenses, prolonging shelf life, and enhancing storage capacity [10]. Drying is a preservation technique that aims to decrease the moisture content of food components, thereby prolonging their shelf life. Nevertheless, dried food components undergo certain modifications, including diminished nutritional value, changes in color, shrinkage, and differences in organoleptic quality, which directly affect consumer acceptability. Conventional drying methods for certain roots and tubers lead to a reduction in the number of bioactive substances present. This is caused by the loss of water, extended processing durations, exposure to oxygen and light, and processing temperatures surpassing 70 °C [11].

Prior to drying, implementing pre-drying process or pretreatment methods can improve permeability, expedite drying rates, stimulate enzymes, and inhibit oxidation in food materials [12]. Deactivating enzymes and reducing moisture using different pretreatment techniques, such as chemical, thermal, and physical approaches [13], are necessary to increase the amount of time tubers can be stored. Chemical precipitation methods can impede enzymatic browning reactions. Frequently employed chemicals for this purpose include sodium metabisulfite, sodium chloride, citric acid, and ascorbic acid [14]. Thermal treatments, such as blanching using hot water and steam, can deactivate polyphenol oxidase (PPO) enzymes. In addition to enzyme deactivation, this method significantly impacts texture, color, flavor, moisture content, and nutritional value [12].

Melese and Keyata [15] and Vera et al. [16] offer useful insights on pretreatment and drying procedures for different types of tubers. However, the cassava tuber drying sector still encounters numerous obstacles. Enduring challenges encompass the intricacy of drying systems, the expensive nature of equipment, and the technical complexities of operations. These obstacles have hindered the progress and acceptance of new drying and pretreatment methods. Therefore, it is crucial to carefully analyze both the economic feasibility and the quality of the output when drying cassava tubers. The objectives of this review were to (i) evaluate various pre-drying techniques and drying methods to understand their effects on drying kinetics and product quality; (ii) identify obstacles and limitations that prevent the production of high-quality cassava flour; and (iii) outline future directions for the pre-drying, drying, and post-drying processes of cassava.

2.2. Developments in Cassava Tuber Pre-Drying and Drying Research

Food drying, along with cooling and freezing, has been a longstanding preservation method. Its primary goal is to reduce moisture content in food products, thus enhancing their shelf life. Various factors influence the drying rate, including water transfer mechanisms, air properties, and material characteristics [17]. Pretreatment, commonly chemical or physical, significantly impacts both the drying process and material quality. One notable effect is the reduction in water activity (aw), which decreases microbial and enzymatic activities, enhancing preservation. Water activity is crucial for determining the availability of water for microbial and enzymatic reactions in food [18]. For staple foods like cassava, understanding the relationship between moisture content and water activity is essential for storage. Cassava sorption isotherms show how moisture content correlates with water activity, guiding optimal drying and storage conditions. Controlling water activity is vital in the drying process, as it affects the removal of water using various techniques. However, drying can impact the texture and quality of the original product, necessitating innovative pretreatment and drying technologies to preserve nutritional content, prevent chemical degradation, and maintain structure and texture [19,20]. By managing water activity during drying, it is possible to achieve preservation goals while preserving sensory and nutritional qualities. This knowledge underscores the importance of considering water activity in preservation strategies to ensure food quality and safety.

Conventional pretreatment and drying methods are being substituted by innovative techniques and hybrid processes, which involve the use of eco-friendly technology to promote sustainable food processing that must be implemented [21]. A survey of the Scopus database was undertaken to explore the progress in the adoption of current and improved procedures. The search was focused on the keywords "cassava + pretreatment" and "cassava + drying" within the time frame of 1990 to 2023. Figure 2.2 displays the quantity and pattern of publication data related to these keywords.

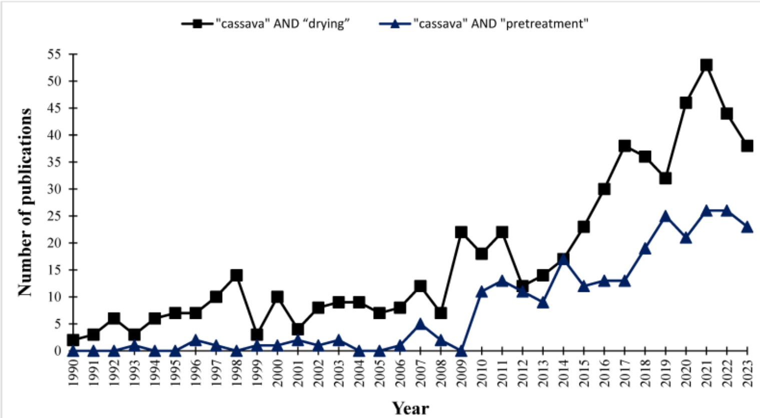

Figure 2.2. Pretreatment and drying technology trends for the period 1990–2023 as measured by the number of publications employing the following keywords: "cassava" AND "pretreatment"; "cassava" AND "drying".

Additionally, safety risks associated with cassava consumption and the drying process are significant factors that warrant attention. Cassava contains cyanogenic glycosides, primarily linamarin, which can release cyanide upon hydrolysis [22]. Improper processing and inadequate drying techniques can lead to high cyanide levels in cassava products, posing health risks such as cyanide poisoning [23]. Moreover, microbial contamination during the drying process can result in the proliferation of harmful pathogens, leading to foodborne illnesses [24]. Therefore, comprehensive evaluations of pretreatment and drying methods for cassava must include assessments of cyanide levels, microbial safety, and overall food safety. Understanding the potential hazards and implementing appropriate control measures are crucial steps in ensuring the quality and safety of dried cassava slices or cassava flour throughout the value chain [25].

2.3. Advancements in the Research of Pre-Drying Techniques for Cassava Tubers

The following section presents a comprehensive overview of recent advancements in the study of pre-drying techniques and other potential techniques applicable to cassava tubers. Pre-drying, whether achieved through chemical or physical means, holds paramount importance in food processing, enhancing drying efficiency and elevating the overall quality of the final product. Physical pretreatment methods, including hot water blanching and steam blanching, play a pivotal role in preserving food quality by focusing on nutrient retention and preventing structural damage. On the other hand, chemical techniques such as acid treatment and sulfiting serve to deactivate enzymes, modify structure, and uphold flavor and nutritional content. Numerous pre-drying processes, particularly conventional pretreatment methods, are undergoing refinement to meet the demands of the lower-to-mid-tier food industry, which necessitates high-quality products with minimal nutrient loss, notwithstanding challenges such as operational inefficiencies and significant product alterations. Table 2.1 elucidates the impact of various pretreatment techniques on the drying characteristics and quality of dried cassava tubers.

Table 2.1. Pre-drying techniques adopted for the drying of cassava tuber.

Pre-Drying Technique	Pre-Drying Condition	Drying Method	Major Observations	References
Hot Water Blanching	70 °C for 6 min Sample-to-water ratio 1:5 (w:w)	Microwave–hot air drying	Pre-drying and drying process had uniform effects on cassava regardless of cultivar variation. Starch changes during cooking did not affect drying kinetics, resulting in consistent texture and sensory scores.	[26]
Steam Blanching	110 °C for 5 min	Solar drying Hot air oven drying Drum drying	Pre-drying with steam blanching followed by drum drying yields higher digestibility and preferable sensory attributes, while hot-air oven drying enhances β-carotene concentration. Cultivar and drying method influence flour characteristics, highlighting the potential of drum drying for optimal product quality.	[27]
Sulfite Solution	0.3% sodium metabisulfite solution for 7 min	Flash drying	Pre-drying with sodium metabisulfite followed by flash-drying enhances rheological properties and water absorption capacity of cassava flour, making it suitable for formulations requiring good pasting quality and moderately high gel strength.	[28]

Acid Solution	0.3% citric acid solution for 7 min	Cabinet drying	Citric acid-treated flour exhibits inferior rheological traits and water absorption capacity compared to sodium metabisulfite-treated counterparts.	[28]
Combined	1.31% citric acid and 1.03% sodium metabisulfite for 20 min Steam blanching at 80 °C for 1.01 min.	Hot-air oven drying	Combined pre-drying conditions enhance cassava drying efficiency, yielding moisture content of 6.19% and whiteness index of 92.00. The logarithmic model best describes dehydration kinetics, crucial for tuber processing improvements.	[29]
Ultrasonic Field	Distilled water with ultrasound (DWU), osmotic dehydration with ultrasound (ODU) Frequency of 20 kHz at ultrasound power Pretreatment time of 600 W and 10 min	Hot-air oven drying	Ultrasound pretreatments, especially ODU, significantly reduce drying time of yellow cassava, enhancing effective moisture diffusivity and reducing energy costs. Cavitation effects create microscopic pathways, influencing both internal and external resistances. Parabolic model fits best for DWU, while Page model suits ODU and untreated samples. Thus, ultrasound pretreatment proves beneficial for hot air drying of yellow cassava.	[30]

2.3.1. Hot Water Blanching

A widely used pretreatment technique before drying is hot water blanching, which involves immersing fresh tubers in hot water at temperatures ranging from 70 to 98 °C for 5 to 15 min [31,32]. The main goal is to preserve the quality of the product by deactivating enzymes, eliminating bacteria, and extracting intercellular air from the tissues [33]. By changing the physical properties of the sample, such as making cell membranes more permeable and creating small cracks on the epidermis, hot water blanching speeds up the drying process [34,35]. However, conventional hot water blanching, despite its uncomplicated equipment and straightforward operation, has certain drawbacks. The pretreatment of cassava slices with hot water blanching at a temperature of 80 ± 2 °C for 5 min yields a lower whiteness index (ranging from 80.48 to 84.05) compared to pretreatment solely through soaking [36]. On the other hand, the degradation of food quality, namely the deactivation of oxidases, can result in alterations in flavor, appearance, and the depletion of heat-sensitive components. Furthermore, it is important to disregard the potential loss of soluble nutrients, such as carbohydrates, minerals, vitamins, sugars, and proteins, which may diffuse into the blanching water [37]. In addition, the process of hot water blanching has a negative impact on the texture and microstructure of the sample [29,38]. In the end, this approach produces a substantial amount of wastewater, which gives rise to environmental problems.

2.3.2. Steam Blanching

Pretreatment steam blanching is used to minimize the loss of nutrients, especially those that dissolve in water, and decrease the number of solid substances that dissolve in water, reducing waste. The selection of this technique is justified in comparison to hot water blanching, as it effectively preserves minerals and water-soluble constituents by minimizing the absorption effect [39]. Steam blanching combined with microwave vacuum drying significantly enhances drying efficiency and color retention of purple-fleshed sweet potato slices. The process reduced drying time to 7 min, achieving 7.5% moisture content. This method offers a rapid drying process with improved color preservation compared to hot water blanching [40]. Steam blanching significantly influences digestibility and β-carotene concentration in dried cassava, with drum-dried flours exhibiting higher digestibility, and air oven-dried samples showing elevated β-carotene levels [27]. However, there are obstacles related to the softening of tissues and changes in quality that occur due to extended heating duration. Additionally, uneven blanching effects occur due to steam condensation on the surface of the product during the initial stage of the process.

2.3.3. Sulfite Solution

The food industry has extensively utilized sulfidation, or the use of sulfur, to minimize browning during drying and to prevent degradation in quality during food processing and storage [41]. The standard method for conducting this procedure involves the utilization of sulfur dioxide gas or water-soluble sulfide salts, such as potassium metabisulfite (K_2S_2O), sodium metabisulfite (Na_2S_2O), and sodium hydrogen sulfide ($NaHSO_3$). Sulfides, when used in low quantities, effectively inhibit enzymatic and non-enzymatic browning, as well as microbiological activity. Sulfite renders PPO inactive by engaging in a chemical interaction with quinones, hence impeding PPO's functionality and diminishing the presence of oxygen [42]. The pretreatment of cassava slices with 4% Na_2S_2O resulted in a moisture content of 6.44% and a whiteness index of 93.87, with the determined activation energy for the process being 32.68 kJ/mol and the calculated effective moisture diffusivity as 6.23×10^{-9} m^2/s [29]. In addition, sulfite functions as an antioxidant to inhibit the degradation of ascorbic acid and safeguard lipids, essential oils, and carotenoids against oxidative harm during processing. This offers the benefits of keeping color, reducing damage, and maintaining some nutritional properties.

Sulfidation can result in the depletion of water-soluble nutritional components, the development of undesirable odors, the alteration of textures to a softer state, and the existence of chemical residues on treated food items [43,44]. While sulfidation is still successful in maintaining the color of products, its drawbacks make it less preferable, particularly in the growing trend of organic food production.

2.3.4. Acid Solution

Acid pretreatment is a prevalent technique that improves the caliber of agricultural commodities by inactivating enzymes, augmenting pigment durability, and altering texture. The basis of this process involves adjusting the pH, inhibiting the activity of polyphenol oxidase, and decreasing color changes caused by enzymatic processes. Acidic substances with a pH between 3.0 and 7.0 are advantageous for retaining the stability of pigments and also help maintain the texture of the product due to their chelating capabilities. Citric acid, a prevalent organic acid, functions as both an anti-browning agent and an efficient texture modifier for fruits and vegetables [45]. The pretreatment of cassava slices with 4% citric acid resulted in a moisture content of 6.53% and a whiteness index of 93.49, with determined activation energy for the process at 32.83 kJ/mol, and an effective moisture diffusivity of 6.32×10^{-9} m^2/s [29].

Although acid pretreatment offers advantages, it also has disadvantages, including the depletion of water-soluble nutrients and the deterioration of acid-sensitive pigments. Nevertheless, this method continues to be valuable for improving the quality and longevity of agricultural goods.

2.3.5. Ultrasonic Field

Ultrasonication is a pretreatment technology that uses high-frequency ultrasonic waves to create, enlarge, and burst bubbles in a liquid medium, a process called cavitation [46]. The pretreatment method, which produces a large number of bubbles, enhances the transfer of mass in food items by utilizing both direct mechanisms (inertial flow and the "sponge effect") and indirect mechanisms (creation of microchannels) [47,48]. The application of ultrasonic treatment at ambient temperature allows for the preservation of thermally delicate components in food [49]. Ultrasonic-assisted techniques, as an emerging treatment, have garnered significant interest in agricultural product research due to their capability to facilitate the drying process, increase drying rates, and enhance product quality by modifying the microstructure of plant tissue [30,31,47,50].

Ultrasound pretreatment of potatoes results in improved color and texture attributes, while it also causes more damage to the cell structure compared to the control group [51]. While it does minimize processing time and preserve product quality, its downside lies in causing structural harm to plant tissues, leading to a reduction in phytochemical content. Technological limitations encompass factors such as restricted capacity to scale up, the intensity of cavitation, and the requirement for coupling media (such as gel, water, or oil), which present difficulties for implementing large-scale applications in the food industry [52]. Possible solutions entail the advancement of non-contact ultrasonic methods that utilize air as a coupling medium to tackle practical challenges and fulfill the requirements of large-scale companies [53].

2.3.6. Alternative Prospective Pre-Drying Techniques for Cassava Tubers

The following section introduces alternative methods for preparing cassava tubers prior to drying, highlighting advancements in their processing techniques. While some approaches show promise in enhancing the efficiency of cassava tuber pretreatment, others may pose challenges such as complexity, high costs, and the need for specialized equipment and expertise. In pursuit of optimizing treatment duration, product quality, and energy consumption, emerging pretreatment systems like pulsed electric field have been explored as potential alternatives to conventional methods such as ethanol pretreatment. These systems often combine multiple pretreatment techniques, leveraging the benefits of each approach. However, selecting the most suitable methods and components requires a comprehensive understanding of the specific requirements and challenges associated with cassava tuber processing. Carvalho et al. [17] have documented various pretreatment technologies for drying roots and tubers, including the effects of ultrasound, pulsed electric field, high hydrostatic pressure and ethanol pretreatment on tubers. Figure 2.3 illustrates the mechanisms of ethanol solution, pulsed electric field, and high hydrostatic pressure pretreatment techniques applied to tubers.

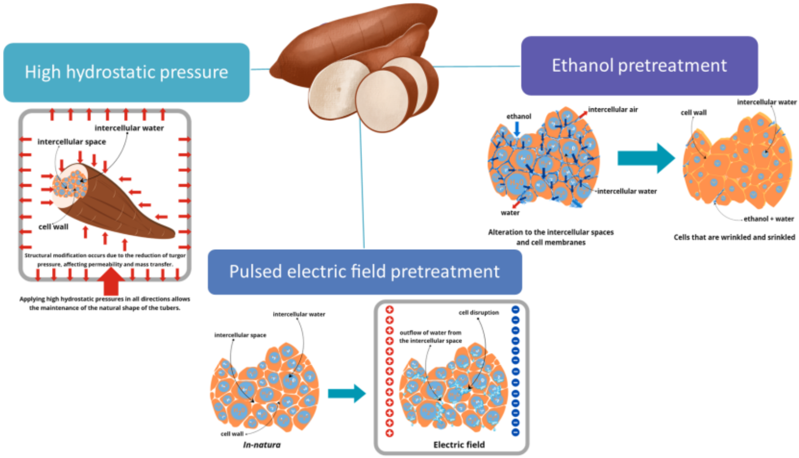

Figure 2.3. The effect of ethanol solution, pulsed electric field and high hydrostatic pressure pretreatment techniques on tubers.

2.3.6.1. Ethanol Solution

The use of ethanol solution as a pretreatment in drying research is becoming more common. It has been found to be effective in different applications, including immersion, spraying, and atmospheric modification of agricultural products [54,55]. The process entails alterations in the structure of plant tissues when exposed to ethanol, such as wilting, thinning of cell walls, and disruption of cellular organization. These changes ultimately facilitate the transfer of mass [56]. Figure 2.3 displays the effects of ethanol pretreatment on tubers. Applying ethanol pretreatment to carrots and potatoes can effectively decrease drying time by up to 50% and significantly reduce energy usage in carrot drying by 56% [57,58].

While the use of this method is successful in decreasing the time it takes for carrots and potatoes to dry, it does not consistently lead to improvements in the overall process. Rojas and Augusto [59] observed that infrared drying of potatoes treated with ethanol negatively impacted their ability to rehydrate due to significant structural alterations. Variables such as the duration of pretreatment and the thickness of the sample have an impact on the extent to which ethanol may penetrate, which is essential for its efficacy, particularly in solid items such as roots and tubers. Although ethanol shows promise, additional research is necessary to maximize its use in improving the drying quality of different types of vegetables, especially roots and tubers.

2.3.6.2. Pulsed Electric Field

Pulsed electric field (PEF) technology is a method that uses short, intense electric pulses to treat food. This treatment can be applied to liquid, semi-solid, or solid food by immersing the food in a liquid before applying the pulses. The treatment is applied between two electrodes [60]. The utilization of PEF involves the inactivation of microbes and enzymes at room temperature while simultaneously maintaining the quality characteristics of food such as color, flavor, texture, and nutritional content [61]. This process improves the ability of substances to pass across the cell membrane and breaks down tissues, hence increasing the movement of substances in plant materials [62]. The impacts of pulsed electric field pretreatment on cassava tubers are illustrated in Figure 2.3. PEF has been successfully used in agricultural products to enhance the drying process by increasing the permeability of cell membranes and protecting food quality by deactivating enzymes [63]. Liu et al. [64] demonstrated that PEF pretreatment in conjunction with vacuum drying effectively reduces the drying process duration, mitigates color alterations, and improves carrot rehydration ability.

Nevertheless, PEF has significant limitations, such as its incapacity to inactivate enzymes in specific circumstances, resulting in cell harm and tissue softening, as well as the considerable expenses associated with the required equipment [65]. Furthermore, the presence of food ingredients that have large particles or exhibit strong electrical conductivity can present difficulties [66].

2.3.6.3. High Hydrostatic Pressure

High hydrostatic pressure (HHP) technology is a type of pretreatment that uses high-pressure shock waves (100–800 MPa) conveyed by water to treat materials at precise durations and temperatures [67,68]. The application of pretreatment in food products leads to cell permeabilization, promotes diffusion, and increases drying rates. The utilization of HHP on cocoyam, Peruvian carrot, and sweet potato has shown decreased drying durations and limited degradation of quality, as indicated by changes in texture, higher rates of moisture transfer, and improved rehydration abilities [69]. Nevertheless, the main obstacles to implementing HHP are the exorbitant expenses associated with the equipment, the limited processing capacity, and the unpredictability of enzyme deactivation [65]. According to Jermann et al. [70], the commercial adoption of HHP in the food industry necessitates further research and development to address structural issues and the permeation of pressure mediums into food products.

2.4. Advancements in the Research of Drying Methods for Cassava Tubers

The following section offers a comprehensive overview of the latest advancements in research pertaining to the drying of cassava tubers, as well as other potential techniques. Drying, a method employed for food preservation since ancient times, is explored in detail here, with a focus on various conventional and emerging methods used for drying cassava tubers. These methods encompass sun drying, solar drying, hot air oven drying, vacuum drying, freeze drying, fluidized bed drying, solar-assisted drying, and microwave-assisted drying. Understanding the strengths and limitations of each technique is crucial for selecting the most suitable approach for specific application scenarios. Table 2.2 outlines the impact of different drying methods on the drying characteristics and quality of cassava tubers.

Drying significantly reduces anti-nutritional factors in cassava. Dahal and Tamang [78] reported that both sun-drying and cabinet drying resulted in substantial reductions in cyanide, tannin, oxalate, and phytate levels compared to raw cassava. Sun-drying, particularly, achieved greater reductions, with cyanide levels decreasing by 56.79% (to 37.29 mg/kg) and tannin, oxalate, and phytate levels decreasing by 94.2%, 79%, and 81.73%, respectively. Cabinet drying also showed reductions, though to a lesser extent, with cyanide decreasing by 23.69% (to 65.84 mg/kg) and tannin, oxalate, and phytate levels decreasing by 12.31%, 37.2%, and 41.34%, respectively. Nebiyu and Getachew [79] similarly reported that sun-drying cassava chips treated with a 24-h water soaking significantly reduced the total hydrogen cyanide content in cassava flour by over 80%.

Mechanical and natural drying of cassava influence anti-nutritional cyanogenic glucoside levels, with sun drying generally leading to better degradation due to optimal enzyme activity and lower temperatures, while oven drying retains more cyanide, especially at higher temperatures and with thinner chips, making drying methods less effective for detoxification of cassava high in initial cyanogen [80]. Drying fresh, peeled cassava chips significantly reduced total cyanogen content, with the extent of reduction influenced by chip size, temperature, and drying time; notably, thicker chips exhibited a higher reduction due to slower drying rates allowing more linamarase–glucoside interaction [81].

Pre-drying fermentation plays a significant role in diminishing cyanide levels in cassava, thereby enhancing linamarase activity [78,82]. This methodology proves highly effective in reducing cyanide content to levels below the recommended threshold prior to the drying process, a departure from conventional techniques. Moreover, the conditions during solar drying exert a notable influence on cyanide retention, implying a versatile strategy to alleviate the presence of anti-nutritional factors in dried cassava [82].

Table 2.2. Comparison of drying and quality characteristics of drying methods for cassava tuber.

Drying Method	Drying Condition	Drying Characteristics and Quality	References
Sun Drying	Temperatures: 18–25 °C; Humidity: 60–75%.	Sun drying significantly influenced the color, proximate, functional, and pasting properties of cassava flour, impacting its quality and industrial applicability. Despite its simplicity and cost-effectiveness, sun drying demonstrates varying effects on different properties compared to other drying methods, emphasizing its importance in cassava processing.	[71]
Solar Drying	Drying time: 40 h; Average drying temperature: 52 °C.	Solar drying of cassava leads to the formation of coarser particles as a consequence of prolonged drying periods and exposure to light, consequently resulting in a reduced β-carotene content. Additionally, this drying method yields fufu with distinctive texture attributes, thereby affecting its stickiness and softness.	[27]
Hot Air Oven Drying	Load: max 120 kg; Drying time: 660 min; Drying temperature: 50, 60, 70 °C;	Hot air oven drying effects on fermented-cooked cassava chips revealed that increasing temperature and fan speed reduced drying time. Effective diffusivity rose with temperature and fan speed, affecting proximate compositions and functional characteristics. Gaussian	[72]

	Fan speed: 0.5, 0.9, and 1.3 m/s.	process regression (GPR)-based modeling proved superior for optimization and control monitoring, vital for product standardization.	
Vacuum Drying	Drying time: 10 h; Drying air temperature 50, 60, and 70 °C; Drying pressure: −60 cmHg.	Vacuum drying significantly influences dried cassava quality. Convective multiple flash drying (CMFD), a novel drying method, achieves desired moisture content in 5–6 h, yielding harder chips compared to convective and vacuum methods. Operating conditions affect moisture content.	[73]
Freeze Drying	Vacuum: Hi MBars pressure, wait for the collector at 22 °C; Run collector: −41 °C for 48 cumulative hours.	Freeze drying significantly preserved color and quality of cassava flour compared to other methods. It yields superior moisture, ash, fat, and protein content, ensuring high-quality flour. Functional properties like swelling power and water absorption are favorable. Pasting properties also indicate its suitability for various food products.	[71]
Fluidized Bed Drying	Drying air temperature: 50, 55, 60, 65, and 70 °C; Solid feed flow rate: 10 g/min and 30 g/min; Airflow rate: 0.012 m^3/min.	In the continuous vibrated fluidized bed drying of cassava starch, air temperature significantly influences drying, while weir height and solid feed rate have minimal impact. The Page model proves most accurate in describing the drying kinetics.	[74]

Sun Drying and Oven Drying	Sun drying for 4–6 h, followed by oven drying at 55 °C for 24 h.	Drying cassava chips followed by milling into flour reduces hydrogen cyanide (HCN) content by up to 81%, enhancing safety. However, slow drying during rainy seasons can lead to higher acidity, compromising taste and favoring spoilage. Whiteness may be affected by ash content and water quality, influencing product quality.	[75]
Solar-Assisted Heat Pump Drying	Load: 30.8 kg; Drying time: 9 h; Drying temperature: 45 °C; Specific moisture extraction ratio (SMER): 0.38 kg/kWh; Coefficient of performance (COP): 3.38; Thermal efficiency (η): 30.9.	Both solar drying (SD) and solar-assisted heat pump drying (SAHPD) significantly reduced cassava mass and moisture content. SAHPD demonstrated higher drying rate, specific moisture extraction rate, thermal efficiency, and pick-up efficiency compared to SD, indicating its superior performance in drying cassava.	[76]
Hybrid Solar Drying	Load: 300 g Drying time: 3 h Drying temperature: 40, 50, 60 °C	Higher drying temperatures lead to faster and more effective drying of cassava starch. The fastest moisture reduction occurs initially, with the drying process mainly happening during the falling rate period. Hybrid solar dryers, especially when combined with liquefied	[77]

		petroleum gas (LPG), are more effective compared to open sun drying, with an effectivity factor reaching up to 6.4.	
Microwave–Hot Air Drying (MHAD)	Drying temperature: 70 °C; Airflow rate: 1.9 m^3 min^{-1}; Microwave power: 95 W.	MHAD affected the physicochemical properties, drying kinetics, and sensory acceptance of dried cassava. MHAD resulted in similar drying kinetics across cultivars, with no significant differences in moisture removal rates.	[26]

2.4.1. Sun Drying

Cassava tubers can be dried either by exposing them directly to sunlight or by placing them on bamboo racks with a plastic sheet underneath. Smallholder farmers widely adopt sun drying due to its straightforwardness and cost efficiency. To expedite the drying process of cassava tubers, it is common practice to decrease their size, hence increasing the exposed surface area [83]. According to Alamu et al. [71], the moisture content of dried cassava tubers can reach a level of 6.55% ± 0.07 by using sun drying. This can be done by maintaining drying settings of 18 to 25 °C and humidity levels between 60 and 75%. Nevertheless, the utilization of open-air drying methods might result in the introduction of impurities and a rise in moisture levels, even after achieving a state of equilibrium moisture content [84]. Alamu et al. [71] also observed that sun drying cassava has the disadvantage of causing the tubers to change color, with the largest ΔE values (16.37–24.78) compared to oven drying. Managing drying parameters in sun drying is a difficult task. Although this process is inexpensive, it lacks reliability in preserving the quality of dehydrated tubers.

2.4.2. Solar Drying

Solar drying is a more advanced drying technique than sun drying, where the drying process takes place inside a mechanical structure that harnesses solar energy [85]. Its operational simplicity and economic affordability have led to its increased appeal, especially among small-scale farmers. Although open-air drying under sunlight is cost-effective and requires minimal work, it has several obstacles. These include extended drying durations, the risk of contamination from stones and debris, and exposure to exhaust emissions on the roadside, which can compromise the nutritional value of the product. According to Suherman et al. [86], solar drying, examined on seaweed and cassava starch, proves effective in reducing moisture content, influenced by drying time and temperature, indicating its viability as a feasible method for efficient drying and maintaining product quality. The yams, which were subjected to solar drying, achieved an equilibrium weight between 390 and 480 min, with drying rates ranging from 10% to 20% per hour [87]. Despite the impact of environmental elements such as temperature, sun radiation, and rainfall on solar drying, several system advancements have been implemented to tackle these challenges.

2.4.3. Hot Air Oven Drying

The process of hot air oven drying is based on the idea of mechanical air convection, where an electrically powered exhaust fan is used to regulate the internal temperature of the drying chamber. The use of hot air ovens for drying yellow cassava yields moderate digestibility levels, ranging from 60.3% to 70.4%, whereas drum drying shows higher digestion rates, ranging from 69.4% to 79.7%, and solar drying yields comparable results, ranging from 60.4% to 70.7%, with hot air oven drying also demonstrating higher concentrations of β-carotene compared to alternative drying techniques [88]. Famurewa et al. [89] found that using a hot air oven at a temperature of 60 °C for a duration of 14 h resulted in cassava tubers being dried more quickly and with a lower moisture content compared to traditional methods like sun drying. The process of drying yams using a hot air oven results in the highest levels of protein, ash, fiber, and carbohydrates, as well as minerals like calcium (Ca), magnesium (Mg), and phosphorus (P). However, the moisture content is generally lower compared to that obtained with sun drying methods [90]. The use of hot air oven drying is favorable due to its cost-effectiveness in comparison to vacuum and freeze-drying methods while still producing higher-quality products than sun drying [71,91].

2.4.4. Vacuum Drying

Vacuum drying is widely used in the food, agricultural, and pharmaceutical industries. It involves drying at low temperatures using steam, hot air, or inert gas. This is done by passing the gas through empty shelves or over hot plates in a high-pressure vacuum environment [92]. Rashid et al. [93] assert that vacuum drying of potatoes is a superior and efficient drying approach when compared to conventional methods like sun drying or open-air drying. This technique expedites the dehydration process, resulting in superior-quality goods and maintaining optimal nutritional value in the dehydrated potatoes. Yang et al. [94] found that vacuum drying of potatoes decreases moisture content, alters starch properties favorably, and reduces oil absorption by forming protective layers, although microstructural changes observed via SEM negatively impact oil absorption, providing insights for improving fried potato quality and controlling oil content. Although vacuum drying has some advantages, it is still very expensive and time-consuming compared to other drying methods mentioned in prior studies. Another obstacle is the ability to scale up this drying process to meet commercial demands.

2.4.5. Freeze Drying

Freeze drying, a technique used to preserve heat-sensitive and biologically based food products for extended periods of time, relies on sublimation as an essential step. Alamu et al. [71] found that freeze drying with a run collector working condition at a temperature of −41 °C for 48 h resulted in sliced cassava tubers with a higher moisture content (9.77 ± 0.01) compared to oven drying (6.22 ± 0.02). Nevertheless, the color alterations observed in the dehydrated cassava tubers using freeze drying exhibited the least magnitude when compared to sun drying and oven drying methods [71]. Various elements, such as the thermal conductivity of the heating plate and the temperature difference between the sample and the air, influence the heat transmission mechanism in freeze drying. Li et al. [95] found that freeze drying preserves appearance, color, microstructure, and active components of Bletilla striata tubers effectively compared to other drying methods, making it the most suitable option for preserving quality and active components, beneficial for medicinal and tonic functions. Freeze drying after high shear mixing (HSM) at varying speeds enhances total dietary fiber content in cassava starch, reaching up to 14.66% at 12,000 rpm, thus improving its potential as a source of resistant starch [96]. In general, the freeze-drying technique for cutting tubers has been thoroughly investigated and proven to be highly effective in creating superior dried products. Nevertheless, this technology has certain disadvantages, such as substantial investment and operational expenses, extended drying durations, and the difficulty of expanding operations.

2.4.6. Fluidized Bed Drying

Fluidized bed drying is based on the principle of sample fluidization, in which moist particles maintain a fluidized state through continuous interaction with a heated surface or hot air blown over them. It is frequently used to remove moisture from wet powders and solid capsules or particles. This technique enables effective heat and mass transmission, leading to quick drying durations, rapid drying rates, effectiveness, and consistent condensation [97]. Furthermore, Famurewa and Emuekele [98] reported that fluidized bed drying at 70 °C and an air velocity of 2.75 m/s significantly reduces the cyanide content in cassava chips. The moisture content of potatoes decreases as the drying air temperature increases while utilizing fluidized bed drying, regardless of whether the potatoes are cuboid or cylindrical in shape [99]. According to Lozano-Acevedo et al. [100], higher temperatures and higher concentrations of citric acid result in more significant alterations in color. Additionally, decreasing the amount of water and water activity in potato samples dried using fluidized bed drying also contributes to these color changes. Fluidization drying systems have wide acknowledgement and effective implementation on an industrial level. Although the distributor placement has certain benefits, such as its static nature and low particle velocity, it can also lead to issues with energy consumption and air circulation. These problems can have a negative impact on drying rates, causing cracking and damage to occur [101].

2.4.7. Solar-Assisted Drying

The solar-assisted heat pump dryer (SAHPD) emerges as a promising technology offering a sustainable solution for drying biomaterials like cassava, thereby mitigating postharvest losses. SAHPDs are favored over other drying methods due to their economic efficiency, reliability, and ability to produce high-quality dried products [102]. Unlike passive solar dryers reliant solely on sunlight or other energy-intensive drying methods, SAHPDs can operate effectively even in the absence of direct sunlight, ensuring consistent product quality throughout the day [103,104]. Loemba et al. [105] reported the significant benefits of utilizing heat pumps in agricultural product drying. Employing heat pumps notably reduced drying time, enhanced specific moisture extraction, and lowered energy consumption. In the case of cassava, the combined use of solar drying (SD) and SAHPD decreased the mass from 30.8 kg to 17.4 kg within 13 and 9 h, respectively, at average temperatures of 40 °C and 45 °C. The moisture content of cassava decreased from 61% to 10.5%, with corresponding mass flow rates [84]. Furthermore, the study reported average thermal efficiencies of 25.6% for SD and 30.9% for SAHPD, along with respective average drying rates and specific moisture extraction rates. The integration of solar-assisted heat pump drying technology exhibits notable improvements in drying characteristics, offering reduced drying times, enhanced energy efficiency, and improved product quality, particularly exemplified in the drying of cassava.

2.4.8. Microwave-Assisted Drying

Conventional microwave drying has a faster drying rate and low energy consumption compared to most other conventional drying methods for drying roots and tubers [17]. Nevertheless, this drying method experiences product non-uniformity as a result of an unequal distribution of the electromagnetic field within the drying chamber. The non-uniform intensity of the electric field, along with insufficient methods to dissipate excessive thermal energy from the product, can cause localized overheating, leading to subpar product quality and tissue harm. In order to overcome these restrictions, researchers have investigated different hybrid microwave drying approaches, including the combination of microwave drying with other methods, such as microwave-vacuum drying (MWVD) [106], microwave-assisted freeze drying (MWFD), and microwave-enhanced spouted bed drying (MWSD), for a range of industrial purposes. According to Regier et al. [107], carrot drying using MWVD required less than 2 h, while conventional drying took 4.5–8.5 h. MWVD has also been observed in the processing of yams [108], potatoes [109], and beets [110]. In a comparison of MWVD, MWFD, and MWSD on carrots, Yan et al. [111] discovered that MWSD produced the most uniform color and the maximum drying rate at 3.5 W/g. Additional research conducted on the drying of potatoes and yams utilizing MWFD also demonstrated enhancements in the drying process, resulting in reduced drying time [108].

2.4.9. Alternative Prospective Drying Methods for Cassava Tubers

This section provides a thorough examination of various research efforts in the field of food drying, with a specific emphasis on novel techniques that can be utilized for drying cassava tubers. Although several drying procedures have proven to be effective in creating dry materials, many of them are complex, expensive, and require specialized equipment and knowledge. In addition, the conversation explores hybrid drying techniques, which researchers have investigated as possible options that provide benefits in terms of drying time, product quality, and energy efficiency compared to traditional methods. Hybrid drying systems usually combine different drying methods to make use of the benefits of each approach. This requires a thorough assessment of criteria and difficulties in order to choose the most suitable techniques and components. In addition to these approaches, various other procedures have been examined for the purpose of drying cassava slices. However, it is crucial to conduct further study and make necessary improvements in order to enhance these techniques. These diverse methods contribute to the ongoing examination and improvement of cassava drying processes, highlighting the significance of continued study and improvement efforts.

2.4.9.1. Infrared-Assisted Drying

Infrared radiation (IR) drying and IR-assisted drying utilize electromagnetic radiation with wavelengths that are longer than those of visible light [21,112]. During the drying process, the material being dried absorbs infrared (IR) energy, resulting in the elimination of moisture and the heating of the substance. This causes the material to dry from the outer surface to the inner part [112]. The heat source can be powered by either electricity or gas, and the radiation strength can be modified to attain the desired degree of drying. Research suggests that infrared (IR) drying improves the quality of dried items by reducing drying time and enhancing rehydration behavior [113]. Nevertheless, the excessive use of electricity can lead to undesirable outcomes in the quality of the end product. This includes reduced ability to rehydrate and noticeable alterations in color for sweet potatoes [114,115], as well as diminished retention of ascorbic acid in carrots [116].

The integration of convective drying and infrared drying techniques yields accelerated drying durations, decreased energy usage, and enhanced mass transfer rates while drying sweet potatoes [115]. However, it is crucial to apply hybrid drying methods correctly in order to prevent oxidative enzymatic activity and the loss of important components in sweet potatoes [117]. Far infrared radiation (FIR) drying has a greater ability to penetrate materials compared to near infrared radiation (NIR). It also has a stronger effect on materials with high moisture content, leading to a higher preservation of antioxidant properties in seaweed compared to freeze drying [118]. In addition, infrared (IR) can also assist in freeze drying processes (IRFD), resulting in reduced drying time and improved quality, as demonstrated in research conducted on sweet potato slices [119]. However, the untapped potential of using IR drying for sliced cassava tubers has not been thoroughly investigated.

2.4.9.2. Ultrasound-Assisted Drying

The incorporation of ultrasonic technology into the HAD process is considered essential to overcome the constraints. Ultrasound technology has potential for improving the HAD process by decreasing the resistance to moisture diffusion within and increasing the transfer of moisture internally [120]. Multiple forms of ultrasound technology, such as air ultrasound and contact ultrasound [36], have been proven to accelerate drying periods and reduce the resistance of moisture transfer throughout the drying process. Mulet et al. [121] state that during the process of drying, mechanical waves travel through the air and food products, causing small waves to form at the surface of the solid. This results in a decrease in external resistance, an increase in mass transfer, and a faster diffusion process. The utilization of ultrasound during the drying process for carrots led to a notable decrease in drying time by 22% when using a power of 200 W [122]. Carcel et al. [123] found similar results, showing that carrots dried 30% faster when subjected to processing conditions of 75 W power and 21.7 kHz frequency. In contrast, the utilization of ultrasound-assisted drying on potatoes resulted in a reduction of drying time by as much as 47.7% when using 60 W power. However, this process did modify the structure of the dried potatoes [124]. The impact of ultrasound differs among various matrices, such as roots and tubers, due to inherent variables including tissue porosity and cell structure compactness. Hence, it is crucial to understand the essential attributes of the food prior to utilizing ultrasound techniques in order to achieve dried food with enhanced quality and reduced drying time.

2.4.9.3. Refractance Window Drying

Refractance window drying (RWD) is a novel drying method that uses thin transparent plastic sheets as a "window" to generate heat through infrared radiation, drying substances as flakes or powder [125]. Subsequently, circulated hot water is used to heat the materials from below. This drying method provides clear benefits, particularly in generating high-quality dried products in terms of color, bioactive components, and overall attractiveness. The drying process for this product is characterized by short duration and low temperatures (30–70 °C), which effectively preserve its nutritional content, including vitamins and antioxidants, as well as its color [126]. Heat transfer takes place through three modes, enabling fast processing, and drying stops once it reaches the point of dry equilibrium [127]. More precisely, RW technology exhibits superior thermal efficiency and cost-effectiveness, as stated by Bernaert et al. [126]. The utilization of RWD has been expanded to encompass a wide range of root crop goods, including potatoes [128]. Duarte-Correa et al. [129] found that potato flour has water-holding capacities ranging from 0.35 to 4.68 g/g and oil-holding capacities ranging from 0.80 to 1.73 g/g. This demonstrates that RW drying is capable of producing powders with reliable and uniform technological properties. As a result, RWD shows great potential for use in various applications within the food industry. Ueda et al. [130] found that the functional, nutritional, and sensory characteristics of the powder produced are impacted by various processing variables, such as water temperature, drying duration, and sample thickness. While RW drying has several benefits, it is important to note that there are potential disadvantages, such as the requirement for meticulous regulation of processing settings and the possibility of impacting the characteristics of the dried product.

2.5. Post-Drying Operations for Dried Cassava Tubers

Cassava, a staple crop in numerous tropical and subtropical regions, holds significant economic importance by serving both as a fundamental food source and as a primary income generator for farmers [131]. Enhancing cassava processing techniques, particularly drying methods, can improve food security, promote sustainable livelihoods, and strengthen social bonds within communities [132]. Notably, advancements in cassava processing, particularly in the realm of drying techniques, have markedly bolstered its market viability and economic worth. This improvement stems from the successful reduction of moisture content to levels compatible with most market requirements [133,134], rendering cassava products stable for extended periods of storage [135].

Cassava chips and cassava flour are the predominant products used in cassava tuber drying applications. The study conducted by Oladejo et al. [30] demonstrated that employing ultrasound in distilled water and an osmotic solution as pretreatment for convective drying of cassava tuber slices might result in significant reductions in energy consumption, time, and overall costs associated with the drying process. Pornpraipech et al. [136] conducted drying research that demonstrated how the shape and weather circumstances affect the drying process of cassava tuber slices. Veeramanipriya and Sundari [137] conducted drying research on cassava tuber slices using a hybrid photovoltaic thermal solar dryer equipped with an evacuated tube collector as the drying processing instrument. The study findings indicated that hybrid drying outperformed sun drying in terms of the dimensions, form, hue, visual aspect, texture, and overall excellence of the dehydrated cassava tuber slices. Assessing modified starch, specifically in flour materials, is essential, particularly due to its involvement in the preservation process, which includes drying [57]. Table 2.3 presents various examples of diverse food products primarily composed of flour that utilize cassava flour.

Effective packaging and storage are essential actions after the drying process to preserve the quality of food products. The presence of carbohydrates, proteins, and lipids in cassava tubers contributes to both enzymatic and non-enzymatic reactions that occur during the storage of dried cassava tubers. Endogenous processes and microbial activity generate a range of aromatic scents and soft textures through biochemical interactions. Cassava tuber slices may harbor harmful microorganisms, such as bacteria, viruses, yeast, and molds. Alamu et al. [71] reported that cassava flour of superior quality is characterized by low ash content and low moisture content.

The storage conditions for cassava flour are a crucial component that directly impacts the quality of the flour for its eventual utilization. Utilizing acceptable packaging materials, such as plastic buckets, jute, low-density polyethylene bags, sacks, and paper bags, is recommended for flour goods [145]. Ogiehor and Ikenobomeh [146] conducted a study where they used different materials, including low-density polyethylene bags, Hessian bags, high-density polyethylene bags, and plastic buckets, to package stored garri at a temperature of 30 °C for 24 weeks. The study found that all packaging types resulted in an increase in moisture content in the samples. Ensuring the ideal temperature and relative humidity is essential for maintaining the quality of products. Inadequate packaging materials can lead to a decline in the quality and shelf life of flour [147]. The choice of packaging materials has an impact on the stability of physicochemical and microbiological properties during storage. According to Opara et al. [147], plastic buckets resulted in the least color change in cassava flour, plastic packaging resulted in the largest amount of carotenoid content, and paper bags resulted in the lowest counts of aerobic mesophilic bacteria and molds in cassava flour.

Efficiently optimizing the drying process is crucial for guaranteeing the exceptional quality of cassava flour. Thus, it is crucial to exert meticulous control over temperature, accurately determine when drying is complete, and maximize drying efficiency. This methodical approach ensures that the used process optimizes the quality of cassava flour. The implementation of cutting-edge measurement and control approaches [148,149], such as low-field nuclear magnetic resonance (LF-NMR) technology, is crucial in achieving this goal. LF-NMR allows for the measurement and tracking of moisture levels during the drying process for cassava tubers. This enables the accurate prediction of when the drying is complete and ensures the production of high-quality cassava flour. These measures not only improve the quality of processing but also reduce costs. To achieve high-quality cassava flour manufacturing, it is essential to optimize the drying process and incorporate LF-NMR technology. Afterwards, the resultant superior cassava tubers can be used together with other items to improve their nutritional content, promoting high-quality growth.

Table 2.3. The utilization of cassava flour in a variety of food products.

Products	Compositions	Key Highlights	References
Biscuits	Cassava flour, full-fat soy-flour, wheat flour, sugar, margarine, egg, baking powder, ginger flour, sugar, margarine and egg	The protein enrichment of cassava flour increased the nutrient content of the biscuits, potentially meeting the nutrient requirements of school children.	[138]
Gluten-free cup cakes	Cassava flour, shortening, egg, milk powder, baking powder and vanilla	The use of cassava flour, along with pumpkin and potato flours, or their mixture, tailored for celiac patients.	[139]
Cookies	Cassava flour, cowpea flour, salt, sugar, skimmed milk, baking fat, and water	A decrease in protein content occurred with the increase in cassava flour substitution, but the addition of cowpea flour led to an overall elevation in protein content.	[140]
Gluten-free flat bread and biscuits	Flat bread: Cassava flour, rice flour, extruded soy protein, xanthan, salt, butter, and water. Biscuits: Cassava flour, rice flour, extruded soy protein, xanthan, sugar, egg, baking powder and butter.	Incorporating cassava flour and extruded soy protein led to an improvement in the nutritional content of both flat bread and biscuits.	[141]

Bread	Cassava flour, wheat flour, fiber, yeast, water, salt and sucrose.	Cassava flour presents a promising solution for replacing up to 30% of wheat flour without significant differences in the final product.	[142]
Bread	Cassava flour, wheat flour, honey, baking fat, yeast, bread improver, water, and salt.	The substitution of sugar with liquid honey in cassava-wheat composite flour formulations led to notable effects on both the pasting properties of cooked dough and the characteristics of the resulting bread.	[143]
Noodles	Cassava flour, water, and alum.	Cassava flour presents a promising solution for replacing wheat flour in noodle manufacturing.	[144]
Noodles	Cassava flour, wheat flour, salt and water.	Noodles made from a composite flour of cassava and wheat showed promising results in terms of acceptability.	[88]

2.6. Conclusions

The increasing demand for cassava and its derivatives requires the implementation of effective pretreatment and drying methods to guarantee high quality and meet the demands of the market. Although these processes are important, there is minimal study on innovative methods compared to those for other dietary components. Future research should prioritize the development of more-efficient arrangements to maintain the nutritional and physicochemical properties, with a specific emphasis on non-thermal pretreatment and low-temperature drying techniques. Furthermore, it is imperative to evaluate the influence of different factors on the quality of cassava flour and develop methods for ensuring quality control and monitoring throughout the pre-drying, drying, and post-drying stages.

The focus should be on implementing effective pretreatment, drying techniques and post-drying operations to decrease operational costs, eliminate waste, and improve profitability for cassava tuber drying facilities. It is important to prioritize the incorporation of pretreatment and drying technologies into small-scale businesses. This can be achieved by focusing on practical challenges through education and awareness initiatives. Furthermore, it is important to create automated technologies for the purpose of process control and quality assessment, while also ensuring a harmonious equilibrium between capital investment and technical advancement. Future study should focus on prioritizing sustainable drying processes for cassava products, taking into account the environmental consequences and societal advantages. Integrating computer-based techniques and artificial intelligence into process control and quality assessment systems is crucial for further enhancing cassava pre-drying, drying, and post-drying technologies. Promising research fields are the enhancement of non-invasive methodologies and the incorporation of artificial intelligence into process management and product evaluation.

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3. Application of Central Composite Design and Superimposition Approach for Optimization of Drying Parameters of Pretreated Cassava Flour.

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Abstract

The primary goals of this study were to identify the influence of temperature and drying time on pretreated cassava flour, as well as the optimal settings for the factors and to analyze the microstructure of cassava flour. The experiment was designed using the response surface methodology with central composite design and the superimposition approach in order to assess the effect of drying temperature (45.85–74.14 °C) and drying time (3.96–11.03 h) and the optimal drying conditions of the cassava flour investigated. Soaking and blanching were applied as pretreatments to freshly sliced cassava tubers. The value moisture content of cassava flour was between 6.22% and 11.07%, whereas the observed whiteness index in cassava flour ranged from 72.62 to 92.67 in all pretreated cassava flour samples. Through analysis of variance, each drying factor, their interaction, and all squared terms had a substantial impact on moisture content and whiteness index. The optimized values for drying temperature and drying time for each pretreated cassava flour were 70 °C and 10 h, respectively. The microstructure showed a non-gelatinized, relatively homogeneous in size and shape sample with pretreatment soaked in distilled water at room temperature. These study results are relevant to the development of more sustainable cassava flour production.

Keywords: blanching; cassava flour; central composite design; soaking; superimposition.

3.1. Introduction

Indonesia is an agrarian nation where the agricultural sector is one of the primary economic development drivers. In Indonesia, numerous agricultural crops are cultivated, including tubers, cereals, legumes, vegetables, and fruits. In 2020, 18.5 million tons of cassava tubers (*Manihot esculenta* Crantz) were the most produced source of carbohydrates other than rice [1]. Indonesia is one of the six largest producers of cassava in the world, along with Nigeria, Democratic Republic of the Congo, Thailand, Ghana, and Brazil [2]. Cassava tubers are the most commonly consumed component of the cassava plant; this part is rich in starch and is the primary storage organ in cassava plants [3,4]. Cassava tubers are one of the most promising agricultural products for diversification into several food varieties. In Indonesia, cassava tubers are processed into a variety of products, including tapioca, mocaf, cassava flour, tapai, chips, and tiwul. Cassava flour is produced from cassava tubers that have been processed using an uncomplicated drying technique [5].

In an effort to expand the use of cassava as a food, postharvest handling and flour processing are employed. Compared to fresh cassava tubers, cassava flour has a longer shelf life and a larger range of applications. The appearance of vascular streaks with bluish-black staining is a sign of postharvest physiological decline in cassava tubers. Microbial activity is the primary cause of cassava tuber destruction [6,7]. Physical (blanching) and chemical (calcium chloride, citric acid, and ascorbic acid) pretreatments are frequently used to prevent the browning and discoloration of tubers caused by enzymatic action. Some of the literature describes the use of blanching as well as ascorbic acid, sulfite, and citric acid in the production of yam flour [8,9]; the use of sulfite in the production of sweet potato starch [10,11]; and the use of calcium chloride treatment in the production of cassava chips [12,13]. However, there are just a few reports using blanching and soaking as the only pretreatments for cassava flour processing.

In recent years, one-factor-at-a-time (OFAT) analysis has been used extensively in the food processing literature, particularly for drying technologies. Statistical analysis and regression coefficient models or the mathematical models are required to predict the process conditions for drying cassava flour. Design of experiments (DOE) provides a number of advantages over conventional analysis, including minimal resource requirements (number of trials, time, materials, and labor), exact prediction findings on the major elements and their interactions, and the capacity to study a large number of factors

[14]. Response surface methodology (RSM) is a statistical technique used to determine the relationship between response variables and a set of input variables [15]. RSM is a statistical and mathematical technique that can be utilized to create, develop, and optimize processes, formulations, or even both [16]. RSM is currently one of the most often used optimization techniques in the world of food technology and engineering. RSM has been used for process optimization in several studies: 1. determine the drying behavior of cassava chips at various temperatures using different cutting shapes [17]; 2. impact of temperature and drying time on the thermal and physical characteristics of cassava flour [18]; 3. as a tool to discover the interactive impact of pretreatment and drying process on the physicochemical of cassava flour [19]; 4. optimization of drying parameters for convective drying and drum drying of sweet potatoes [20,21].

There is currently a lack of information regarding the influence of blanching and soaking on cassava flour when the drying process (temperature and time) is optimized using RSM, particularly the central composite design (CCD) and superimposition approach. As pretreatments in this investigation, soaking in distilled water and blanching were applied separately. The objectives of this study were to: (i) investigate the effect of drying parameters on the moisture content (*MC*) and whiteness index (*WI*) of cassava flour; (ii) carry out optimization, verification, and superimposition processes to achieve the optimal combination of factors that generate minimum *MC* and maximum *WI* of cassava flour; and (iii) analyze the microstructure of cassava flour and evaluate the results.

3.2. Materials and Methods

3.2.1. Design of Experiment Based on RSM

The software Design Expert version 13.0.5.0 (Stat-Ease Inc., Minneapolis, MN, USA) was used to construct an experimental matrix for processing samples of cassava flour. When designing experiments with RSM, there were two drying parameters that served as the basis: the drying temperature (T_1) and the drying time (T_2). The three pretreatments were applied independently, and then each treatment was processed with the T_1 and T_2 configurations according to the experimental matrix. As for the responses of the two factors, which are the moisture content (*MC*) and whiteness index (*WI*) of cassava flour. Table 3.1 shows the five specified levels and operating ranges for the CCD.

Table 3.1. Factors and levels used for *MC* and *WI* analysis.

Factor	**Unit**	**Notation**	**Level**				
			−1.414	**−1**	**0**	**1**	**1.414**
Temperature	°C	T_1	45.8579	50	60	70	74.1421
Time	Hours	T_2	3.9644	5	7.5	10	11.0355

3.2.2. Experiment Design

Based on the five levels, two factors, and three replications applied to all design points, the CCD developed by Design Expert software (Stat-Ease Inc., Minneapolis, MN, USA) generated a total of 39 experiments. These variables were chosen because they have a considerable impact on the responses and the permissible working range, as documented in the literature. Table 3.2 displays the full CCD, including both coded and uncoded factor values. The total value of the block is 1 and the experiments are conducted in a random order.

Table 3.2. Design matrix of the experiment.

Sample			Coded Factor		Uncoded Factor	
Pretreatment A	Pretreatment B	Pretreatment C	T_1	T_2	T_1	T_2
A1	B1	C1	−1	−1	50	5
A2	B2	C2	−1	−1	50	5
A3	B3	C3	−1	−1	50	5
A4	B4	C4	1	−1	70	5
A5	B5	C5	1	−1	70	5
A6	B6	C6	1	−1	70	5
A7	B7	C7	−1	1	50	10
A8	B8	C8	−1	1	50	10
A9	B9	C9	−1	1	50	10
A10	B10	C10	1	1	70	10
A11	B11	C11	1	1	70	10
A12	B12	C12	1	1	70	10
A13	B13	C13	−1.414	0	45.8579	7.5
A14	B14	C14	−1.414	0	45.8579	7.5
A15	B15	C15	−1.414	0	45.8579	7.5
A16	B16	C16	1.414	0	74.1421	7.5
A17	B17	C17	1.414	0	74.1421	7.5
A18	B18	C18	1.414	0	74.1421	7.5
A19	B19	C19	0	−1.414	60	3.9644
A20	B20	C20	0	−1.414	60	3.9644

A21	B21	C21	0	−1.414	60	3.96 44
A22	B22	C22	0	1.414	60	11.03 55
A23	B23	C23	0	1.414	60	11.03 55
A24	B24	C24	0	1.414	60	11.03 55
A25	B25	C25	0	0	60	7.5
A26	B26	C26	0	0	60	7.5
A27	B27	C27	0	0	60	7.5
A28	B28	C28	0	0	60	7.5
A29	B29	C29	0	0	60	7.5
A30	B30	C30	0	0	60	7.5
A31	B31	C31	0	0	60	7.5
A32	B32	C32	0	0	60	7.5
A33	B33	C33	0	0	60	7.5
A34	B34	C34	0	0	60	7.5
A35	B35	C35	0	0	60	7.5
A36	B36	C36	0	0	60	7.5
A37	B37	C37	0	0	60	7.5
A38	B38	C38	0	0	60	7.5
A39	B39	C39	0	0	60	7.5

The significance of the main components and their interactions was determined using an analysis of variance (ANOVA) with a significance threshold of 95% and a p-value of 0.050. The mathematical models were derived from the ANOVA table. These models were then used for optimization purposes, the outcome of which was determined by the value of the correlation coefficient, R^2. The experimental data were fitted to a second-order polynomial model to generate a regression coefficient model. Equation (1) illustrates the model form for response surface analysis:

$$Y = \beta_0 + \sum_{t=1}^{3} \beta_i X_i + \sum_{i}^{3} \beta_{ii} X_i^2 + \sum_{i-1}^{2} \sum_{j=i+1}^{3} \beta_{ij} X_i X_j \qquad (1)$$

where Y is the response, β_0, β_i, β_{ii}, and β_{ij} are the regression coefficients for the intercept, linear, quadratic, and interaction, respectively. X_i and X_j are coded values in independent variables [22].

3.2.3. Raw Materials

The tubers of cassava were purchased in a local market in the village of Pasar Laguboti, which is located in the Laguboti District of the Toba Regency in the province of North Sumatra, Indonesia. The local farmers in the village harvested cassava tubers 13 to 17 months after planting. The cassava tubers were sorted before being cleaned in order to eliminate soil and prevent contamination during processing. To minimize injury to the tubers, processing occurs only after 24 h have passed since their collection [13].

3.2.4. Processing of Pretreated Cassava Flour

The procedure described by the Indonesian Agency for Agricultural Research and Development [23] is modified for the processing of cassava flour. The modification of the procedure includes pretreatments consisting of blanching and soaking each experimental sample in distilled water. After cleaning the cassava tubers, they were manually peeled and sliced into 3 × 3 × 1 ± 1 cm (length × width × thickness) pieces. Freshly sliced cassava tubers were subjected to three pretreatments: A (blanched at 80 ± 2 °C for 5 min then soaked in distilled water for 48 h), B (soaked in distilled water for 48 h then blanched at 80 ± 2 °C for 5 min), and C (soaked in distilled water for 72 h at room temperature, 24 ± 4 °C). The cassava slices were then dried in a drying machine (400 W Food Dehydrator, ATHOME collection, West Jakarta, Indonesia) according to the experimental matrix at the temperature and time stated (Table 3.2). The parameters for drying in this study were drying temperature (45.85–74.14 °C) and drying time (3.96–11.03 h). A dry milling machine (HR 2115 Dry Mill Blender, PT. Philips Batam, Batam, Indonesia) was utilized to process the dry chips. The flour obtained from the mill was sieved and kept at room temperature in a plastic sample bag until further analysis.

3.2.5. *MC* Analysis

The *MC* of cassava flour was calculated using standard analytical chemistry procedures [24]. The percentage of *MC* is expressed on a dry basis using the following Equation (2):

$$MC\ (\%) = \frac{W_t\ (\mathrm{g}) - W_i\ (\mathrm{g})}{W_i\ (\mathrm{g})} \times 100 \qquad (2)$$

where, *MC* is moisture content; W_t is the weight of the sample at time t; and W_i is the weight of the sample after drying to a constant weight.

3.2.6. Color Measurement

Using a colorimeter (CS-10, Hangzhou Caipu Technology Co., Ltd., Hangzhou, China), samples of cassava flour were measured in three repetitions. The instrument was calibrated using a bright white standard reference tile and a bright black standard reference tile. During color assessment, L* (brightness), a* (positive values indicate redness and negative values indicate greenness), and b* (positive values represent yellowness and negative values represent blueness) values were collected. According to Torbica et al. [25], the value of the *WI* can be quantitatively determined by combining the L*, a*, and b* components into a single computed term. The formula for *WI* can be found as follows:

$$WI = 100 - \sqrt{a^{*2} + b^{*2} + (100 - L^{*})^{2}} \qquad (3)$$

3.2.7. Microstructure Analysis

Utilizing a scanning electron microscope (SEM) (EVO MA10, Carl Zeiss Pvt. Ltd., Oberkochen, Germany), morphological structural analysis was performed with the purpose of determining the effect of pretreatments (A, B, and C) and drying parameters (T_1 and T_2) on the structures of cassava flour particles. Double-sided tape was used to adhere the samples to the bronze visualization portions. A thin layer of gold was coated on the surface of the sample using a sputter period of 60 s and a sputter power of 20 mA. Surface pictures were captured using an SE (secondary electron) detector with a working distance (WD) of 11.5–12 mm and an extra-high-tension (EHT) of 11.0 kV at 1000× magnification for all samples.

3.3. Results and Discussion

Table 3.3 displays the design configuration derived from the Design Expert program as well as the experimental responses data (*MC* and *WI*). Temperature and drying time are two experimental design variables represented by T_1 and T_2, respectively.

Table 3.3. Design matrix and response value for *MC* and *WI* tests.

Sample	Response		Sample	Response		Sample	Response	
	MC (%)	*WI*		*MC* (%)	*WI*		*MC* (%)	*WI*
A1	13.21	83.17	B1	13.58	80.28	C1	9.51	90.98
A2	13.12	83.53	B2	13.78	79.86	C2	9.26	91.45
A3	12.82	83.06	B3	14.07	80.59	C3	9.88	91.18
A4	12.46	83.81	B4	14.03	80.78	C4	9.57	91.35
A5	12.75	83.36	B5	13.62	81.05	C5	9.21	91.82
A6	13.04	83.44	B6	13.56	80.38	C6	9.14	90.87
A7	12.33	82.68	B7	13.15	79.41	C7	9.18	90.04
A8	12.48	82.53	B8	13.78	79.78	C8	9.45	90.25
A9	12.87	82.15	B9	13.26	79.21	C9	9.52	90.14
A10	10.07	83.44	B10	10.64	80.55	C10	6.22	91.42
A11	10.13	83.65	B11	10.72	80.22	C11	6.51	91.57
A12	10.28	83.37	B12	10.56	80.78	C12	6.62	91.32
A13	13.23	82.13	B13	14.07	79.24	C13	9.58	90.22
A14	13.27	82.17	B14	13.94	79.18	C14	10.02	90.18
A15	13.39	82.15	B15	14.12	79.28	C15	9.65	90.13
A16	10.84	83.29	B16	11.62	80.57	C16	7.23	90.82
A17	11.13	83.36	B17	11.86	80.45	C17	7.61	91.46

A18	10.72	83.29	B18	11.82	80.45	C18	7.78	91.62
A19	13.32	84.05	B19	14.35	81.13	C19	9.88	92.25
A20	12.88	84.05	B20	14.42	81.27	C20	10.13	91.72
A21	12.86	84.14	B21	14.56	81.15	C21	9.96	92.07
A22	10.82	83.07	B22	11.42	80.22	C22	7.34	91.58
A23	11.12	83.18	B23	11.74	80.21	C23	7.52	91.62
A24	11.07	83.24	B24	11.66	80.24	C24	7.65	91.46
A25	11.15	80.56	B25	11.58	78.16	C25	7.45	88.87
A26	11.12	80.83	B26	11.85	77.58	C26	7.45	89.17
A27	11.18	80.62	B27	11.75	77.95	C27	8.06	88.72
A28	11.55	80.91	B28	11.52	77.92	C28	7.73	88.56
A29	11.07	81.14	B29	12.03	77.72	C29	7.65	88.58
A30	11.12	80.71	B30	11.72	77.67	C30	7.58	88.61
A31	11.26	80.48	B31	11.73	77.71	C31	8.16	89.15
A32	10.83	81.06	B32	11.52	77.76	C32	7.86	88.68
A33	11.34	81.20	B33	12.07	77.74	C33	7.72	88.70
A34	10.83	80.61	B34	11.54	77.62	C34	7.61	88.64
A35	10.84	80.76	B35	11.07	77.67	C35	8.03	88.61
A36	10.87	80.82	B36	11.87	77.81	C36	7.54	88.82
A37	10.91	81.18	B37	11.74	77.80	C37	7.56	89.12

A38	11.12	80.59	B38	11.63	78.24	C38	7.87	88.68
A39	11.14	80.61	B39	11.56	78.06	C39	7.94	88.94

3.3.1. Statistical Analysis of *MC*

According to the results of the ANOVA shown in Table 3.4, all of the primary factors (T_1 and T_2) are highly significant at a p-value of 0.000. The coefficients of determination (R^2) of the samples with pretreatments A, B, and C, respectively, are 0.9624, 0.9713, and 0.9648. They indicate that the *MC* in each sample A, B, and C is correlated to T_1 and T_2 by 96.24%, 97.13%, and 96.48%, respectively. If R^2 equals 1, it indicates that the regression coefficient model can predict the optimal value with a high degree of accuracy. The p-value obtained for the lack of fit test was not statistically significant for all pretreatment samples. The high value of the regression and the statistically insignificant lack of fit indicate that the model fits the data well when it is applied.

Table 3.4. *MC* for different T_1 and T_2.

Source	Notation	Sum of Squares	Mean Square	Coefficient	Standard Error	*p*	R^2	R^2 (adj)
Pretreatment A								
Constant				11.0887	0.0560	0.000	0.9624	0.9567
Temperature	T_1	13.93	13.93	−0.7618	0.0443	0.000		
Time	T_2	13.20	13.20	−0.7415	0.0443	0.000		
Temperature∗time	T_1*T_2	3.31	3.31	−0.5250	0.0626	0.000		
Temperature∗temperature	T_1*T_1	5.71	5.71	0.5230	0.0475	0.000		
Time∗time	T_2*T_2	4.82	4.82	0.4805	0.0475	0.000		
Lack of fit		0.2347	0.0782			0.172		
Error		1.32	0.0440					
Total		41.30						
Pretreatment B								
Constant				11.6787	0.0572	0.000	0.9713	0.9669
Temperature	T_1	13.72	13.72	−0.7562	0.0453	0.000		
Time	T_2	21.22	21.22	−0.9402	0.0453	0.000		
Temperature∗time	T_1*T_2	5.40	5.40	−0.6708	0.0640	0.000		
Temperature∗temperature	T_1*T_1	7.41	7.41	0.5959	0.0485	0.000		
Time∗time	T_2*T_2	8.98	8.98	0.6559	0.0485	0.000		

Lack of fit		0.1800	0.0600			0.310		
Error		1.44	0.0481					
Total		56.47						
Pretreatment C								
Constant				7.7473	0.0573	0.000		
Temperature	T_1	14.89	14.89	−0.7878	0.0453	0.000		
Time	T_2	16.04	16.04	−0.8175	0.0453	0.000		
Temperature∗time	T_1*T_2	5.43	5.43	−0.6725	0.0640	0.000	0.9648	0.9595
Temperature∗temperature	T_1*T_1	4.10	4.10	0.4430	0.0485	0.000		
Time∗time	T_2*T_2	5.09	5.09	0.4938	0.04854	0.000		
Lack of fit		0.0955	0.0318			0.604		
Error		1.53	0.0509					
Total		46.12						

Factor interactions (T_1*T_2) and all squared terms (T_1*T_1 and T_2*T_2) are statistically significant at a p-value less than 0.050. Due to the largest absolute coefficient value, primary factors (T_1 and T_2) are seen to have the highest impact on the response for all sample pretreatments. The significant (p-value 0.000) squared term indicates that the interaction between factors and responses follows a curved line. The Equations (4)– (6) present the regression coefficient model of pretreatments A, B, and C, respectively, for the several variables that contribute to the *MC* of cassava flour:

$$Y_{MC} = 11.0887 - 0.7618(T_1) - 0.7415(T_2) + 0.5230(T_1)^2 + 0.4805(T_2)^2 - 0.5250(T_1)(T_2) \quad (4)$$

$$Y_{MC} = 11.6787 - 0.7562(T_1) + 0.9402(T_2) + 0.5959(T_1)^2 + 0.6559(T_2)^2 - 0.6708(T_1)(T_2) \quad (5)$$

$$Y_{MC} = 7.7473 - 0.7878(T_1) + 0.8175(T_2) + 0.4430(T_1)^2 + 0.4938(T_2)^2 - 0.6725(T_1)(T_2) \quad (6)$$

where Y_{MC} represents *MC* as the response, whereas T_1 and T_2 are the temperature and drying time, respectively. This mathematical model can be used to determine and assess the impact of variables on the *MC* of cassava flour.

3.3.2. Effect of Factors on *MC*

The impact of T_1 and T_2 on the *MC* of cassava flour was determined using ANOVA and regression coefficient models based on statistical analysis. Figure 3.1 illustrates the effect of temperature and drying time on the *MC* of cassava flour with a 3D surface graph. Drying conditions with low *MC* were detected at drying temperatures of 70 °C for 10 h for all pretreated samples. The lowest observed concentration of *MC* in cassava flour treated with C was 6.22%. Temperature and time are among the most critical elements that directly influence the drying kinetics during thermal drying.

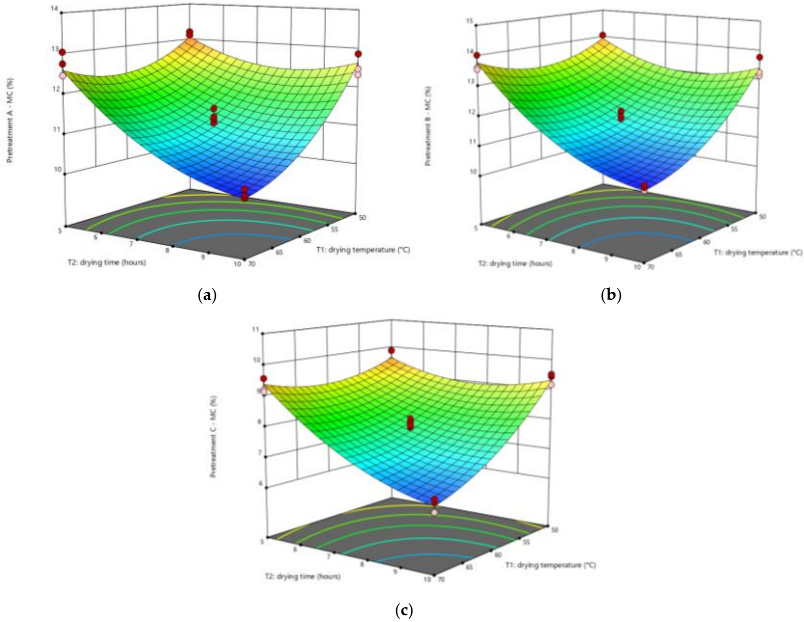
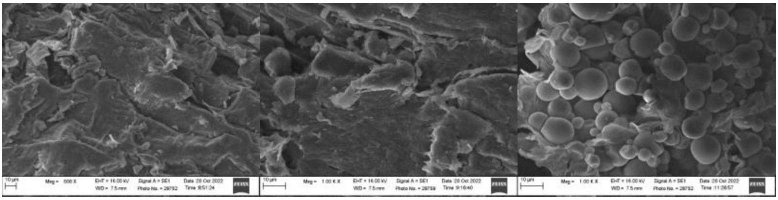


Figure 3.1. Response surface plot for *MC* of cassava flour with pretreatment (**a**) A; (**b**) B; (**c**) C. The blue, green, yellow, and red colors on the surface represent the gradient range from the lowest to the greatest response value, respectively. The red dot represents the response value above the surface, while the pink dot represents the response value below the surface.

Blanching is accomplished by applying an instant and modest thermal treatment to the sample. Enzymatic inactivation, physical structure alteration, and flavor and nutritional content preservation are all targets [26,27]. The serial soaking–blanching–boiling of cassava chips produced a higher drying rate and lower moisture desorption [27]. The *MC* of cassava flour ranged from 10.07% to 13.29% in samples with pretreatment A, between 11.07% and 14.07% in samples with pretreatment B, and between 6.22% and 10.13% in samples with pretreatment C. The *MC* of samples prepared with blanching was higher than that of samples not pretreated with blanching under the same drying conditions. This phenomenon arises due to the fact that blanching promotes starch gelatinization and that during the subsequent drying process, a barrier layer forms on the surface of the sample, which minimizes the amount of water that is transferred from the sample to the atmospheric air [28,29]. Ai et al. [30] also reported that higher heating slowed the drying process and lengthened the dehydration period. Similar findings were discovered by Chen et al. [31], who discovered that the *MC* in unblanched samples of yam flour was lower than blanched samples of the flour. They found that the water-binding capacity (WBC) value of the blanched samples was higher compared to the unblanched samples of yam flour. According to Tacer-Caba et al. [32], higher blanching temperatures and other thermal operations lead to a greater degree of starch gelatinization. The degree of gelatinization and starch fragmentation are the two most important factors influencing WBC [33].

Figure 3.2 depicts a microscopic picture of the A, B, and C samples, which were processed at 70 °C for 10 h. Oval and spherical granules were observed in samples treated with C. The sample granules that followed the blanching procedure presented a variety of forms and sizes, with some of them having been gelatinized. The granules represented in Figure 3.2c are non-gelatinized and relatively homogeneous in size and shape. Figure 3.2a, b show some of the granules that have been gelatinized into enormous masses with block-like and irregular structures as well as voids and rough surfaces. These results are the consequence of the partial gelatinization and subsequent retrogradation of starch appearing to be held together by binding factors such as water and gelatinized starch [34,35].



(**a**) (**b**) (**c**)

Figure 3.2. Microstructure of cassava flour with pretreatment: (**a**) A; (**b**) B; and (**c**) C at 1000× magnification after being dried at 70 °C for 10 h.

3.3.3. Statistical Analysis of *WI*

As can be seen in Table 3.5 of the results of the analysis of variance (ANOVA), the findings revealed that all of the primary factors (T_1 and T_2) were extremely significant with a p-value of 0.000. The coefficients of determination of the samples with pretreatments A, B, and C, respectively, are 0.9774, 0.9772, and 0.9657. They indicate that the *WI* in each sample A, B, and C is correlated to T_1 and T_2 by 97.74%, 97.72%, and 96.57%, respectively. If the value of R^2 is 1.0000, then this can be taken as the ability of the regression coefficient model to accurately predict the optimum value

Table 3.5. *WI* for different T_1 and T_2.

Source	Notation	Sum of Squares	Mean Square	Coefficient	Standard Error	*p*	R^2	R^2 (adj)
Pretreatment A								
Constant				80.8053	0.0529	0.000	0.9774	0.9739
Temperature	T_1	3.29	3.29	0.3702	0.0418	0.000		
Time	T_2	1.73	1.73	−0.2683	0.0418	0.000		
Temperature∗time	T_1*T_2	0.4219	0.4219	0.1875	0.0592	0.003		
Temperature∗temperature	T_1*T_1	19.42	19.42	0.9646	0.0449	0.000		
Time∗time	T_2*T_2	41.47	41.47	1.4096	0.0449	0.000		
Lack of fit		0.1154	0.0385			0.449		
Error		1.27	0.0424					
Total		61.24						
Pretreatment B								
Constant				77.8273	0.0547	0.000	0.9772	0.9737
Temperature	T_1	4.13	4.13	0.4150	0.0432	0.000		
Time	T_2	2.08	2.08	−0.2942	0.0432	0.000		
Temperature∗time	T_1*T_2	0.2324	0.2324	0.1392	0.0611	0.029		
Temperature∗temperature	T_1*T_1	21.15	21.15	1.0068	0.0463	0.000		
Time∗time	T_2*T_2	42.53	42.53	1.4276	0.0463	0.000		

Lack of fit		0.0798	0.0266			0.639		
Error		1.40	0.0466					
Total		64.75						
Pretreatment C								
Constant				88.79	0.0656	0.000	0.9657	0.9605
Temperature	T_1	3.43	3.43	0.3782	0.0519	0.000		
Time	T_2	0.9848	0.9848	−0.2026	0.0519	0.004		
Temperature∗time	T_1*T_2	0.9919	0.9919	0.2875	0.0734	0.004		
Temperature∗temperature	T_1*T_1	17.55	17.55	0.9171	0.0556	0.000		
Time∗time	T_2*T_2	43.25	43.25	1.4396	0.0556	0.000		
Lack of fit		0.3597	0.1199			0.130		
Error		1.77	0.0591					
Total		62.08						

Factor interactions (T_1*T_2) and all squared components (T_1*T_1 and T_2*T_2) are statistically significant at a p-value less than 0.050. The squared factors (T_1*T_1 and T_2*T_2) had the most impact on the response, as indicated by the highest absolute coefficient value of 0.9171 to 1.4396. T_1*T_2 obtained a p-value of 0.003, 0.029, and 0.004, respectively, for the samples with pretreatment A, B, and C for the interaction between the two factors, indicating that there is a significant association between the two factors. The squared term reveals that the relationship between the factors and the responses forms a curved line, and its significance is demonstrated by the fact that the p-value is less than 0.050. The regression coefficient model for the parameters influencing the *WI* of cassava flour is shown in Equations (7)– (9) for the sample with pretreatments A, B, and C, respectively.

$$Y_{WI} = 80.8053 + 0.3702(T_1) - 0.2683(T_2) + 0.9646(T_1)^2 + 1.4096(T_2)^2 + 0.1875(T_1)(T_2) \quad (7)$$

$$Y_{WI} = 77.8273 + 0.4150(T_1) - 0.2942(T_2) + 1.0068(T_1)^2 + 1.4276(T_2)^2 + 0.1392(T_1)(T_2) \quad (8)$$

$$Y_{WI} = 88.79 + 0.3782(T_1) - 0.2026(T_2) + 0.9171(T_1)^2 + 1.4396(T_2)^2 + 0.2875(T_1)(T_2) \quad (9)$$

Y_{WI} represents the response for *WI*, whereas T_1 and T_2 represent the temperature and drying time, respectively. Calculating and analyzing the influence of various factors on the *WI* of cassava flour is possible with the help of these regression coefficient models. The mathematical model demonstrates that the p-value of the lack of fit test and the regression value of the model are progressively high and insignificant. The non-significant lack of fit and high regression value indicate that the implemented model is well-fitting.

3.3.4. Effect of Factors on *WI*

In terms of customer preference for the physical quality of food, color is a crucial component, particularly with regard to flour-based products. Morrot et al. and Zellner & Durlach [36,37] reported that drying circumstances altered the color of various agricultural products. Temperature and drying time are responsible for the discoloration caused by thermal and oxidation reactions during drying [38,39,40].

Cassava flour with acceptable physical and color qualities is white flour. Akintunde and Tunde-Akintunde [41] similarly reported low a* values (−0.07–7.50) and b* values (4.92–8.99) and high L* values (52–80.02) for cassava starch and yam flour, which is consistent with the findings of this study. However, the modest variances in L*, a*, and b* values can be related to changes in the varieties that were utilized and the drying procedures that were used. *WI* reflects the degree of whiteness of food products and the extent of color transformation during food processing [42]. The analysis of the 3D surface graph depicting variations in *WI* angles under different drying conditions of flour indicates that cassava drying at the temperatures and time ranges used in this study can assist in preserving the color of cassava flour, thereby increasing consumer acceptance, utilization, and application in the food industry.

Figure 3.3 depicts the 3D surface graphs illustrating the impact of T_1 and T_2 on *WI*. The *WI* of cassava flour ranged from 80.48 to 84.05 in samples with pretreatment A, between 77.62 and 81.27 in samples with pretreatment B, and between 88.56 and 92.07 in samples with pretreatment C. The highest *WI* values were found in samples pretreated with C that dried at 60 °C for 3.96 h. This could imply that blanching cassava tubers for 5 min at 80 ± 2 °C in hot water was sufficient to drive an increasing non-enzymatic browning reaction. Quayson et al. [43] reported that non-enzymatic browning intensities of yam decreased as soaking time increased. They also discovered that as blanching time increased, non-enzymatic browning levels increased. According to a study done by Sanful et al. [44], samples that were not pretreated showed higher L* values than those that had been blanched in yam flour. Figure 3.4 displays the cassava flour produced under drying conditions of 70 °C for 10 h. As seen in the picture, cassava flour treated with pretreatment C is whiter than cassava flour treated with pretreatments A and B. The photos represent the *WI* value, which indicates that cassava flour with pretreatment C has the highest *WI* value among the others.

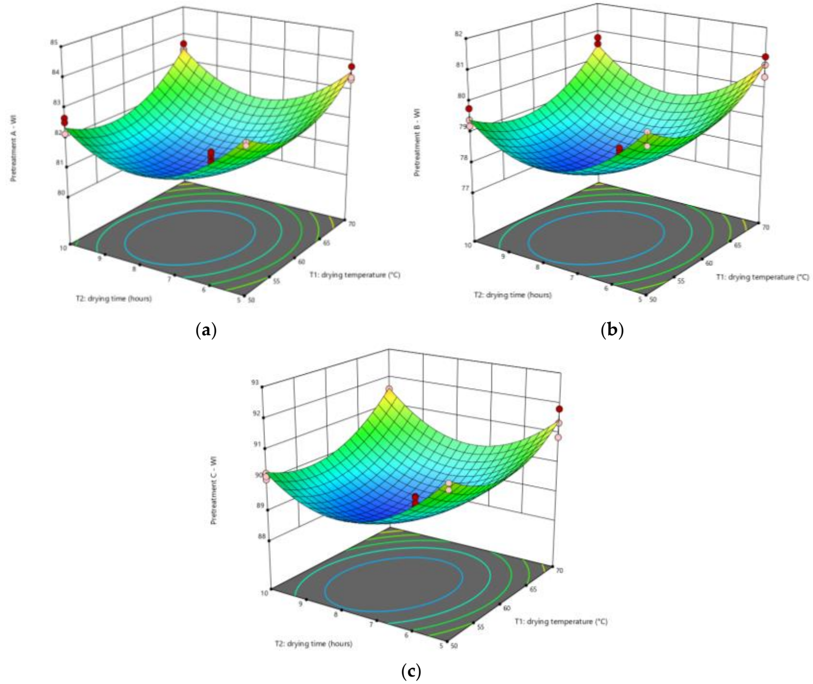


Figure 3.3. Response surface plot for *WI* of cassava flour with pretreatment (**a**) A; (**b**) B; (**c**) C. The blue, green, yellow, and red colors on the surface represent the gradient range from the lowest to the greatest response value, respectively. The red dot represents the response value above the surface, while the pink dot represents the response value below the surface.

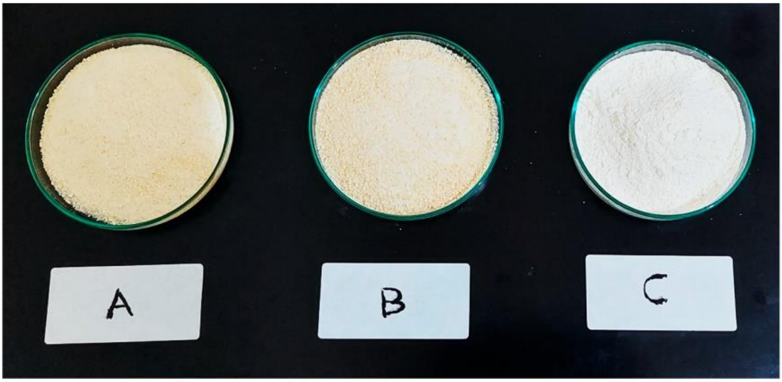


Figure 3.4. The pretreated cassava flour after being dried at 70 °C for 10 h.

3.3.5. Optimization of *MC* and *WI*

The optimization process was conducted to determine the optimal temperature and drying time for producing cassava flour with the lowest *MC* and highest *WI* values. All factors were within the workable range because the desired composite value, D, was calculated to be close to 1. The D values of cassava flour with pretreatments A, B, and C, respectively, were 0.90, 0.89, and 0.89. Figure 3.5 displays the cassava flour optimization plot for all pretreated cassava flour. The optimal values for T_1 and T_2 for all pretreated cassava flour were 70 °C and 10 h, respectively. Cassava flour with pretreatment A had an *MC* of 10.06% and a *WI* of 83.47 in the optimum drying parameters, whereas cassava flour with pretreatment B had an *MC* of 10.63% and a *WI* of 80.52. Cassava flour with pretreatment C had the lowest *MC* (6.41%) and the highest *WI* (91.61) compared to the other pretreatments in the optimum drying conditions. These findings are consistent with those obtained in other investigations, which found a minimum MC and maximum *WI* in each type of processed cassava flour.

Omolola et al. [18] reported that the *WI* and L* of the cassava flour samples were relatively high. Flour typically has an MC of less than 12% [45]. Furthermore, a low moisture content is required to limit microbial growth in food [46].

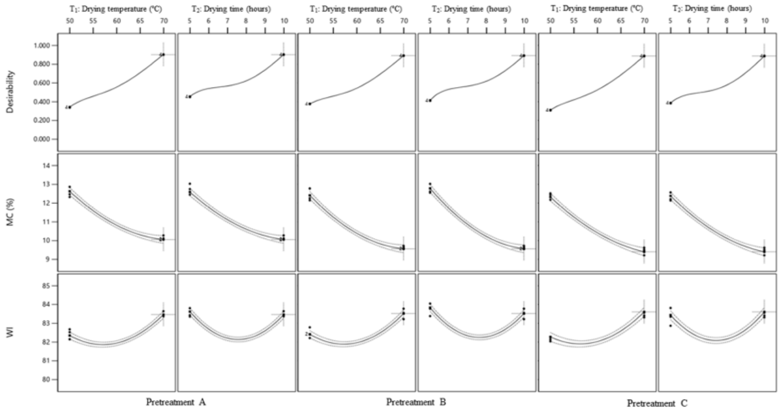


Figure 3.5. Optimization plot for the optimum T_1 and T_2.

3.3.6. Experimental Verification

Experimental verification is the final phase in the modelling procedure and is used to check that the predicted model (the regression coefficient model) is accurate [47]. The experiment was conducted under optimal conditions derived from the optimization plot, with three replicates of each sample. According to the data presented in Table 3.6, the mean relative deviations for *MC* and *WI* were, respectively, 1.48% and 0.12% for samples that had been subjected to pretreatment A; 1.48% and 0.16% for samples that had been subjected to pretreatment B; and 1.29% and 0.16% for samples that had been subjected to pretreatment C. By comparing the experimental (actual) value to the predicted figures, this verifies the predictability of the model and indicates that the RSM-based empirical model can accurately explain the correlation between the variables and the goal response, thereby successfully confirming the optimal process conditions. The *MC* of cassava flour samples processed under varied drying validation conditions ranged from 7.43% to 10.50%, whereas *WI* values ranged from 80.38 to 91.83. According to Onitilo et al. [48], the percentage *MC* of cassava flour ranges from 3.59% to 11.53%, and these results fall within that range. Similarly, the *WI* follows the same pattern as the L* value. Omolola et al. [18] recorded cassava flour *WI* values between 82.88 and 89.42.

Table 3.6. Experiment Verification.

Sample	***MC* (%)**			***WI***		
	Predicted	Actual	Relative Deviation (%)	Predicted	Actual	Relative Deviation (%)
Pretreatment A						
AV1	10.06	10.12	0.59	83.47	83.62	0.18
AV2	10.06	10.23	1.68	83.47	83.35	0.14
AV3	10.06	10.28	2.16	83.47	83.43	0.05
	Mean		1.48	Mean		0.12
Pretreatment B						
BV1	10.56	10.36	1.91	80.52	80.68	0.20
BV2	10.56	10.71	1.68	80.52	80.38	0.17
BV3	10.56	10.47	2.16	80.52	80.61	0.11
	Mean		1.48	Mean		0.16
Pretreatment C						
CV1	6.41	6.37	0.63	91.61	91.42	0.21
CV2	6.41	6.54	2.01	91.61	91.57	0.04
CV3	6.41	6.49	1.24	91.61	91.83	0.24
	Mean		1.29	Mean		0.16

3.3.7. Contour Plots Superimposition

The superimposition of contour plots is the approach used to plot overlay graphs for diverse response surfaces. This technique is superior to the conventional OFAT approach, which does not account for the interaction between the selected variables and involves complex experiments [49]. The overlay contour plot functions as a convenient template for evaluating the response for every given factor value within the defined range. The optimal range of achievable drying settings for pretreating cassava flour is represented in Figure 3.6. Based on the contour plots that were superimposed, the ideal range for the minimum *MC* values and the maximum *WI* values was determined to be 70 °C and 10 h for all pretreatments. The grey areas represent the optimal drying area for all pretreated cassava flour samples.

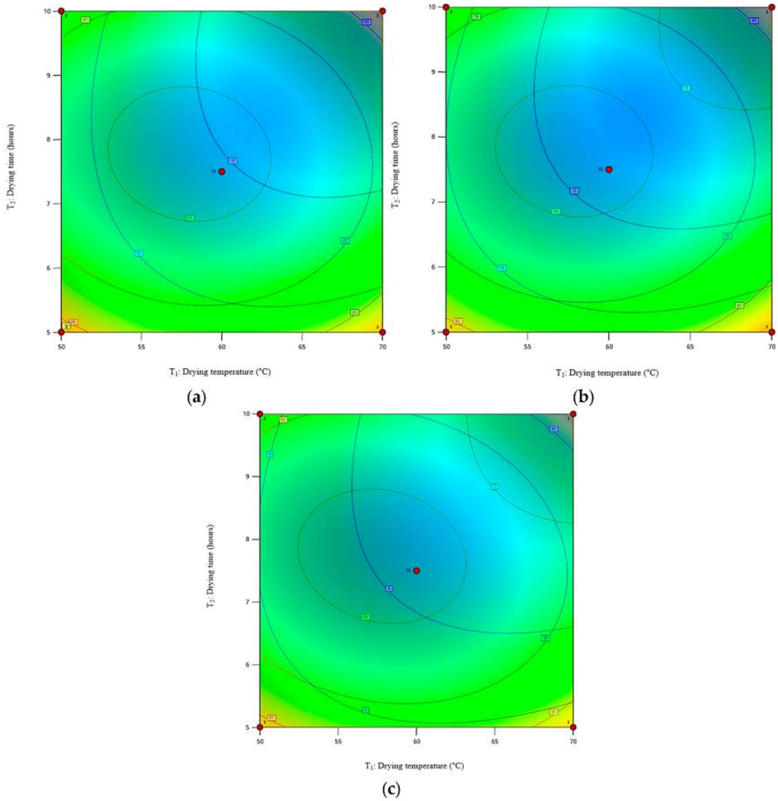


Figure 3.6. Superimposition of the contour plots for the optimum drying conditions for cassava flour with (**a**) A, (**b**) B, and (**c**) C pretreatments.

3.4. Conclusions

The impact of temperature and drying time on the moisture content and whiteness index of each pretreated cassava flour has been examined. Temperature and drying time had a substantial impact on pretreated cassava flour's *MC* and *WI*, as shown by statistical analysis utilizing RSM and CCD. In all experimental designs, the lowest *MC* of cassava flour was between 6.22% and 11.07%, whereas the greatest observed *WI* in cassava flour ranged from 72.62 to 92.67 in all pretreated cassava flour samples. The microstructure revealed that the highest *MC* sample featured starch gelatinization, and a barrier layer formed on the surface of the sample during the drying process. The thermal processing of cassava tubers led to a greater degree of starch gelatinization.

The constructed prediction models, or the regression coefficient models, proved to be highly accurate. The superimpositions of the contour plots were successfully expanded to pinpoint the optimum area of drying parameters for the minimum *MC* and maximum *WI* values, which were identified under process conditions of 70 °C and a drying duration of 10 h for all pretreated cassava flour samples. According to the validation results, the average relative deviation for the *MC* and *WI* ranged from 0.12% to 1.48%.

There are a number of possible research projects that have been explored, including the cassava flour drying kinetics model. Furthermore, studies on the interaction between pretreatment and drying conditions, in addition to other drying methods, have the potential to increase the quality of cassava flour.

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4. Chemical and Thermal Treatment for Drying Cassava Tubers: Optimization, Microstructure, and Dehydration Kinetics.

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Abstract

Perishable commodities like cassava necessitate effective postharvest preservation for various industrial applications. Hence, optimizing pretreatment processes and modeling drying kinetics hold paramount importance. This study aimed to optimize cassava pretreatment using the central composite design of a response surface methodology while also assessing microstructure and dehydration kinetics. Diverse chemical and thermal pretreatments were explored, encompassing sodium metabisulfite concentrations (0–4% w/w), citric acid concentrations (0–4% w/w), and blanching time (0–4 min). The four investigated responses were moisture content, whiteness index, activation energy (E_a), and effective moisture diffusivity (D_{eff}). Employing five established drying models, suitability was appraised after optimal pretreatment conditions were determined. The findings revealed that moisture content ranged from 5.82 to 9.42% db, whereas the whiteness index ranged from 87.16 to 94.23. D_{eff} and E_a ranged from 5.06×10^{-9} to 6.71×10^{-9} m^2/s and 29.65–33.28 kJ/mol, respectively. The optimal pretreatment conditions for dried cassava were identified by optimizing the use of 1.31% citric acid, 1.03% sodium metabisulfite, and blanching time for 1.01 min. The microstructure indicated that particular chemical and thermal pretreatment configurations yielded particles in the shape of circular and elliptical granules. The logarithmic model provided the most accurate description of the dehydration kinetics, with the highest R^2 value (0.9859) and the lowest χ^2, *RMSE*, and *SSE* values of 0.0351, 0.0015, and 0.0123, respectively.

Keywords: cassava tubers; chemical treatment; dehydration kinetics; microstructure; optimization; thermal treatment.

4.1. Introduction

The postharvest processing of cassava tubers plays a vital role in determining the overall quality and safety of the commodity. This phase serves as an intermediary stage between the raw materials and the final products. Clearly, the objectives of the Millennium Development Goals for sustainability align with the imperative to ensure food safety and combat poverty. Postharvest processing plays a crucial role in extending the storage life of highly perishable commodities, including vegetables, fruits, and tubers such as cassava. The exploitation of cassava in downstream activities is crucial, since it provides chances for lucrative market endeavors [1].

Cassava (Manihot esculenta) is a tuberous plant belonging to the Euphorbiaceae family. The total global cassava production in the year 2020 amounted to 302 million tons, with Africa accounting for 64% of the production, Asia contributing 27%, the Americas providing 8.9%, and Oceania contributing 0.1% [2]. Cassava is widely used as a primary dietary source in tropical countries, making it a staple meal for a significant portion of the population. Its substantial potential in the food business has also been acknowledged by researchers [3]. The moisture level of freshly harvested cassava tubers generally varies from 60% to 72.60% [4-6]. The presence of elevated levels of moisture in tubers has a detrimental impact on their shelf life, transportation, and marketability, leading to substantial losses during the post-harvest period. The studies conducted by Riley et al. [7] and Srikanth [8] revealed that an elevated moisture content in yam tubers resulted in microbial spoilage, accelerated degradation, and triggered chemical and biological responses. The utilization of cassava tubers through various drying methods is crucial in order to effectively tackle these difficulties.

The drying process incorporates heat and mass transfer, which play a significant role in diminishing the moisture content of raw materials and modifying the physicochemical characteristics of the resulting dried product [9,10]. The process of reducing moisture in food involves the application of heat to facilitate the movement of moisture from the inside to the surface of the food material. This is subsequently followed by diffusion and evaporation processes [11]. According to Kerkhof and Coumans [12], the energy consumption attributed to drying procedures in industrialized nations ranges from 7% to 15%. The drying procedures employed in food production can consume significant amounts of energy and have a notable influence on the overall quality of the resulting dried food product [13]. The pivotal importance of mass and heat transfer properties in biomaterials is evident in the context of drying, which necessitates the implementation of pretreatment strategies to mitigate energy consumption in the drying process. Various chemical and physical treatments, including soaking in distilled water, blanching, chemical immersion, and salt immersion, have been employed on different tubers prior to the drying process [14-17].

Additional variables that impact the rate of drying encompass the speed at which air velocity, the air temperature, and the relative humidity. The factors mentioned in this context are connected to the effective moisture diffusivity and activation energy of a food material. Effective moisture diffusivity is a crucial transport property that depends on both the drying conditions and the characteristics of the material [18]. According to Rafiee et al. [19], effective moisture diffusivity must be taken into account when designing moisture transfer procedures for food product drying. Additionally, the concept of activation energy is employed to characterize the level of ease associated with moisture transfer in these food items. The activation energy necessary for commencing the drying process, as well as representing moisture diffusion throughout drying, can be estimated using the Arrhenius equation [20,21].

The establishment of kinetic models pertaining to the drying process of cassava tubers is of paramount importance in comprehending their characteristics and dynamics throughout the drying procedure. Mathematical models can be utilized to make predictions about the patterns of moisture transfer in biomaterials. Moreover, mathematical models play a crucial role in the design of drying systems and the analysis of heat and mass transfer events. A correlation exists between the parameters of a simulation model and the conditions of the drying process. It has been found that semi-theoretical models are good for describing how food items dry and for measuring how much moisture is removed during drying processes [11,22]. In addition, the utilization of a drying model offers advantages pertaining to the preservation of products and subsequent food processing [23]. In the study conducted by Cosme-De Vera et al. [24], it was found that the utilization of mathematical models in the context of drying may effectively capture and explain the variability observed in experimental data. Furthermore, the application of these models can lead to a reduction in experimental errors, optimization of drying processes, reduction in energy consumption, and ultimately, an enhancement of profitability [25-29].

The stability of food items can be improved through the implementation of drying techniques, as the reduction in moisture content has a significant impact on biological activity and the occurrence of physical and chemical alterations throughout the storage period [30]. The drying time has a significant impact on the amount of energy needed [11]. The length of drying can be influenced by various aspects, such as the initial treatment of the material, the temperature at which drying takes place, the features and size of the material, and the type of containers used for drying [11,22]. The optimization of the pretreatment entails the formulation of parameters and the selection of ideal factor combinations in order to minimize energy consumption during drying processes and attain the most favorable outcomes. Chemical concentration, soaking time, blanching duration, and blanching temperature are often employed factors in the chemical and thermal preparation of tubers [31-34]. Response Surface Methodology (RSM) is a statistical technique employed to optimize pre-drying treatments. RSM has been utilized as an optimization technique in the context of chemical and physical treatments given to cassava tubers [35-38].

Previous research has looked at how to improve chemical and physical pretreatment methods for cassava tubers. This study, on the other hand, uses response activation energy and effective moisture diffusivity as its main variables. Hence, there is a scarcity or absence of available knowledge pertaining to the optimization of pretreatment procedures utilizing chemical and thermal techniques, specifically in terms of their impact on moisture content, whiteness index, activation energy, and effective moisture diffusivity. The primary aims of this study encompass three key objectives. Firstly, to enhance the parameters involved in the pretreatment processes of cassava tubers. Secondly, to conduct a comprehensive analysis and evaluation of the microstructure of cassava flour obtained through pretreatment, utilizing both chemical and thermal methodologies. Lastly, to assess and determine the most suitable model for the drying kinetics of cassava tubers, considering the optimal conditions achieved through pretreatment.

4.2. Materials and Methods

4.2.1. Sample Preparation

Freshly harvested mature cassava tubers were purchased from local farmers at the Laguboti Market in the Laguboti District of the Toba Regency in North Sumatra Province, Indonesia. Malang 4 was the variety of cassava used in this study due to its cultivation development in the regency. Prior to the pretreatment and drying procedures, the cassava tubers were sorted according to their size and the extent of physical injury, washed with clean (clear, odorless, and purified) water, peeled, and sliced vertically across their dimensions to a thickness of approximately 5 mm using a knife. To minimize injury to the tubers, sample preparation occurred within 24 h of cassava tuber collection [39].

4.2.2. Pretreatment and Drying Experiments

Various pretreatments were then applied to the cassava slices. The moisture content of fresh cassava slices was measured to be 68.31% ± 0.37. The experimental design for pretreatment involved three independent variables: sodium metabisulfite concentrations (0–4% w/w), citric acid concentrations (0–4% w/w), and blanching times (0–4 min). A total of 20 experimental variations were generated from the three levels and three factors using central composite design (CCD) with Design Expert 13.0.5.0 software (Stat-Ease Inc., Minneapolis, MN, USA). Each run was repeated three times, and the mean values of the data collected from these repetitions were recorded. The chemical treatment consisted of soaking cassava slices in various concentrations of sodium metabisulfite and citric acid solutions for 20 min. In the meantime, the thermal treatment consisted of variable durations of 80 °C steam blanching.

The treated samples were drained and then dried in a thin layer using a forced convection drying machine (400 W Food Dehydrator, ATHOME collection, West Jakarta, Indonesia) operating at an air velocity of 2 m/s. The drying temperature was set at 70 °C, following the methodology of a prior study [16]. The sample weight was monitored at 30-minute intervals and considered constant when three consecutive measurements showed no variation, indicating that a stable moisture content had been achieved under the drying conditions. The cassava slices were then milled with a dry milling machine (HR 2115 Dry Mill Blender, PT. Philips Batam, Batam, Indonesia) and sieved using an 80-mesh sieve prior to being preserved at room temperature in plastic sample bags for subsequent analysis. Four responses were investigated, including moisture content, whiteness index, activation energy, and effective moisture diffusivity.

4.2.3. Moisture Content Analysis

The moisture content of the dried samples was assessed by measuring the beginning and final moisture content of the samples using an oven-drying technique in accordance with the Association of Official Analytical Chemists (AOAC) [40] guidelines. The percentage of moisture content was determined on a dry basis using Equation (1).

$$MC\ (\%) = \frac{W_t\ (g) -\ W_i\ (\mathrm{g})}{W_i\ (g)} \times 100 \qquad (1)$$

In this context, *MC* represents the percentage of moisture content, W_t denotes the weight of the sample at a specific time t, and W_i signifies the weight of the sample after drying to a constant weight.

4.2.4. Color Measurement

The color measurements of the samples were performed in this investigation using a colorimeter (CS-10, Hangzhou Caipu Technology Co., Ltd., Hangzhou, China). The equipment underwent calibration by utilizing standard white and black reference tiles prior to conducting measurements. The parameters that were assessed encompassed L* (representing brightness), a* (representing redness, where positive values indicate red and negative values indicate green), and b* (representing yellowness, where positive values indicate yellow and negative values indicate blue). The integration of these values was performed using the whiteness index (*WI*) Formula (2), as established in the work conducted by [41], in order to assess the whiteness index of cassava flour.

$$WI = 100 - \sqrt{a^{*2} + b^{*2} + (100 - L^*)^2} \qquad (2)$$

4.2.5. Determination of Effective Moisture Diffusivity

The effective moisture diffusivity of cassava slices is determined by applying Fick's second law of diffusion to slab geometry, as indicated by Equation (3). Subsequently, Equation (4) was converted into logarithmic form to facilitate the estimation of the moisture diffusivity (D_{eff}) value.

$$MR = \frac{M_i - M_e}{M_o - M_e} = \frac{8}{\pi^2} \sum_{n-1}^{\infty} \frac{1}{(2n+1)^2} exp\left(\frac{-(2n+1) \times \pi^2 \times D_{eff} \times t}{4H^2}\right) \qquad (3)$$

$$Ln(MR) = Ln\left(\frac{8}{\pi^2}\right) - \left(\frac{\pi^2 \times D_{eff} \times t}{4H^2}\right) \qquad (4)$$

where *MR* represents the dimensionless moisture ratio, M_i is the moisture content at *i* time (% db), M_e is the equilibrium moisture content (% db), M_o the initial moisture content (% db), *H* is half of the slices thickness (m), *n* is a positive integer, D_{eff} represents the effective moisture diffusivity (m^2/s), ratio *t* is the time of drying (min). Equation (4) gives the linear representation of Fick's law. Consequently, a graph of *Ln (MR)* versus time produced a slope equal to $\frac{\pi^2 \times D_{eff}}{4H^2}$, thereby determining the effective moisture diffusivity as described by [42,43].

4.2.6. Determination of Activation Energy

The following Equation (5) represents the calculation of the activation energy using the Arrhenius equation:

$$D_{eff} = D_o exp\left(-\frac{E_a}{RT_a}\right) \quad (5)$$

where E_a represents the activation energy (kJ/mol), R represents the universal gas constant (8.3143 J/mol/K), T_a represents the absolute air temperature (K) and D_o represents the pre-exponential factor from the Arrhenius equation (m^2/s).

4.2.7. Microstructure Analysis

A scanning electron microscope (SEM) (JSM-6510LA, Jeol Ltd., Tokyo, Japan) was utilized to examine the morphology of both the untreated sample and the sample treated with chemical, thermal, and their combined methods. The objective of the microstructure analysis was to assess the impact of pretreatments on the particle morphology of cassava flour. The effects of pretreatment configurations on the response, specifically moisture content, can be ascertained by examining the morphological structure. The images in the scanning electron microscope instrument were captured with an applied voltage of 10 kV.

4.2.8. Statistical Analysis

Randomizing the experimental order completely prevented systematic errors in the statistical analysis of this study. Response analysis used a number of statistical tools, such as determination coefficient (R^2), adjusted determination coefficient (adj R^2), coefficient of variation (CV), lack of fit, and analysis of variance (ANOVA). Response surface methodology devised with central composite design using Design Expert version 13.0.5.0 (Stat-Ease Inc., Minneapolis, MN, USA) was used for modeling and optimizing the pretreatment process. The process of optimization consisted of endeavors to minimize all parameters, including citric acid concentration, sodium metabisulfite concentration, and blanching time.

4.2.9. Dehydration Kinetics

Once the optimal pretreatment conditions were determined, drying experiments were carried out to determine the actual drying behavior of cassava slices. The moisture ratio (*MR*) was determined by utilizing the simplified Fick's diffusion law, as expressed in Equation (3). The collected data were then statistically analyzed utilizing five commonly used thin-layer drying models, as presented in Table 4.1.

Table 4.1. Existing mathematical drying models.

Model's Name	Model	Reference
Henderson and Pabis	$MR = a \exp(-kt^n)$	[44]
Page	$MR = \exp(-kt^n)$	[45]
Logarithmic	$MR = a \exp(-kt) + c$	[46]
Newton	$MR = \exp(-kt)$	[47]
Wang and Singh	$MR = 1 + at + bt^2$	[48]

Using Minitab® 19.1 (Minitab LLC, Pennsylvania, PA, USA) software, experimental data obtained for the optimized moisture ratio were subjected to nonlinear regression analysis using the selected drying model parameters in order to evaluate the model parameters. The sufficiency of the data fit was examined by analyzing four statistical parameters: root mean square error (*RMSE*), coefficient of determination (R^2), sum of square error (*SSE*), and reduced chi-square (χ^2). These parameters are represented by Equations (6)– (9).

$$R^2 = \frac{1 - [\sum_{i=1}^{n}(MR_{pre,i} - MR_{exp,i})^2]}{[\sum_{i=1}^{n}(MR_{pre,i} - MR_{exp,i})^2]} \quad (6)$$

$$SSE = \frac{\sum_{i=1}^{n}(MR_{pre,i} - MR_{exp,i})^2}{N} \quad (7)$$

$$\chi^2 = \frac{\sum_{i=1}^{n}(MR_{pre,i} - MR_{exp,i})^2}{N - z} \quad (8)$$

$$RMSE = \left[\frac{\sum_{i=1}^{n}(MR_{pre,i} - MR_{exp,i})^2}{N}\right]^{\frac{1}{2}} \quad (9)$$

where N represents the quantity of experimental units, $MR_{pred,i}$ represents the predicted values, $MR_{exp,i}$ represents the experimental moisture ratio values, *MR* represents the predicted moisture ratio, and z represents the drying model's constant values. Higher R^2 values, lower *SSE*, lower χ^2 values, and lower *RMSE* values were used to evaluate the quality of fit [49-51].

4.3. Results and Discussion

4.3.1. Pretreatment Parameters of Dried Cassava

The design configuration and experimental response data (moisture content, whiteness index, activation energy, and effective moisture diffusivity) are presented in Table 4.2. The three factors included in the experimental design are the concentration of citric acid, sodium metabisulfite, and the duration of blanching.

Table 4.2. Design matrix and responses of dried cassava.

Sample	Uncoded and Coded Factors			Responses			
	SM (% *w*/*w*)	*CA* (% *w*/*w*)	*BT* (min)	*MC* (% db)	*WI*	D_{eff} (m^2/s) × 10^{-9}	E_a (kJ/mol)
S1	0 (−1)	0 (−1)	0 (−1)	7.81 ± 0.13	91.86 ± 0.06	5.81 ± 0.08	33.28 ± 0.43
S2	4 (+1)	0 (−1)	0 (−1)	6.44 ± 0.04	93.87 ± 0.05	6.23 ± 0.03	32.68 ± 0.38
S3	0 (−1)	4 (+1)	0 (−1)	6.53 ± 0.17	93.49 ± 0.07	6.32 ± 0.04	32.83 ± 0.56
S4	4 (+1)	4 (+1)	0 (−1)	6.17 ± 0.09	94.19 ± 0.03	6.52 ± 0.07	32.16 ± 0.44
S5	0 (−1)	0 (−1)	4 (+1)	9.42 ± 0.14	87.16 ± 0.08	5.06 ± 0.09	31.86 ± 0.25
S6	4 (+1)	0 (−1)	4 (+1)	8.79 ± 0.09	87.74 ± 0.05	5.55 ± 0.02	31.44 ± 0.39
S7	0 (−1)	4 (+1)	4 (+1)	8.86 ± 0.12	87.49 ± 0.02	5.47 ± 0.05	31.62 ± 0.47
S8	4 (+1)	4 (+1)	4 (+1)	9.18 ± 0.15	88.13 ± 0.08	5.37 ± 0.07	31.16 ± 0.52
S9	0 (−1)	2 (0)	2 (0)	6.17 ± 0.18	90.39 ± 0.04	6.49 ± 0.04	30.51 ± 0.24
S10	4 (+1)	2 (0)	2 (0)	5.85 ± 0.22	90.96 ± 0.03	6.71 ± 0.04	29.84 ± 0.35
S11	2 (0)	0 (−1)	2 (0)	6.36 ± 0.14	90.22 ± 0.06	6.48 ± 0.05	30.36 ± 0.27
S12	2 (0)	4 (+1)	2 (0)	6.14 ± 0.11	90.75 ± 0.07	6.39 ± 0.08	30.15 ± 0.43
S13	2 (0)	2 (0)	0 (0)	6.15 ± 0.06	94.23 ± 0.04	6.32 ± 0.06	32.64 ± 0.16
S14	2 (0)	2 (0)	4 (+1)	8.22 ± 0.08	89.84 ± 0.08	5.34 ± 0.03	30.85 ± 0.23
S15	2 (0)	2 (0)	2 (0)	6.46 ± 0.13	91.28 ± 0.03	6.31 ± 0.05	29.74 ± 0.22
S16	2 (0)	2 (0)	2 (0)	6.32 ± 0.09	90.83 ± 0.06	6.34 ± 0.08	29.83 ± 0.46

S17	2 (0)	2 (0)	2 (0)	6.13 ± 0.08	90.62 ± 0.09	6.47 ± 0.04	29.65 ± 0.27
S18	2 (0)	2 (0)	2 (0)	6.41 ± 0.05	91.07 ± 0.05	6.34 ± 0.03	30.04 ± 0.34
S19	2 (0)	2 (0)	2 (0)	5.96 ± 0.16	90.89 ± 0.02	6.44 ± 0.07	29.85 ± 0.18
S20	2 (0)	2 (0)	2 (0)	5.82 ± 0.12	91.35 ± 0.06	6.62 ± 0.05	30.18 ± 0.26

Note: values reported are means ± SD. *SM*, sodium metabisulfite concentrations; *CA*, citric acid concentrations; *BT*, blanching time; *MC*, moisture content; *WI*, whiteness index; D_{eff}, effective moisture diffusivity; E_a, activation energy.

4.3.1.1. Moisture Content

Table 4.2 displays the percentage of moisture content in both untreated and treated cassava flour. In the presence of chemical pretreatment, the moisture content typically decreases, ranging from 5.82% to 9.98%. Figure 4.1 provides additional detail regarding the impact of chemical and thermal pretreatment on the percentage of moisture content. The findings of plot 4.1a indicate that an elevated concentration of chemical pretreatment has a discernible impact on the reduction in moisture content in the absence of blanching pretreatment. Increasing the blanching time (2 min) and sodium metabisulfite concentration (4% w/w) leads to better results in lowering the moisture content of dried cassava, as shown in Figure 4.1b. Previous studies reported comparable outcomes in yam samples subjected to sulfiting as a pretreatment [9,52]. In contrast, it was observed that the samples subjected to a blanching duration of 4 min exhibited the greatest levels of moisture content, which varied between 8.22% and 9.18%. This is due to the fact that blanched samples have higher values for water-binding capacity (WBC) than unblanched samples [53]. WBC values are predominantly influenced by the extent of starch fragmentation and gelatinization in the food product [54].

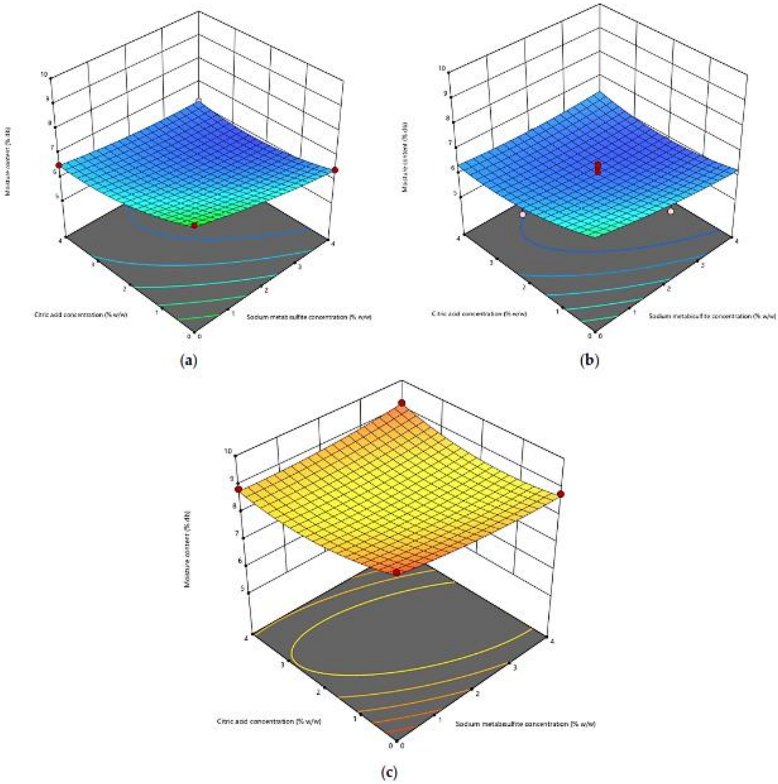

Figure 4.1. Impact of sodium metabisulfite concentration (X1) and citric acid concentration (X2) on the moisture content (Y) of cassava slices blanched for (a) 0 min, (b) 2 min, (c) 4 min.

The utilization of citric acid and sodium metabisulfite in the chemical treatment of cassava tubers affects the moisture content of the dried cassava tubers. Table 4.2 demonstrates that the cassava tubers treated with citric acid have a higher moisture content compared to the tubers treated with sodium metabisulfite. This is consistent with the findings reported by Ngoma et al. [31], who discovered that sweet potatoes that were pretreated with citric acid had a higher moisture content compared to those pretreated with sodium metabisulfite. This related to the ability of sodium metabisulfite to enhance the removal of water from tuber slices by causing modifications in the permeability of cellular membranes [55].

4.3.1.2. Whiteness Index

The role of color in flour-based food products is of great significance, as it significantly influences customer preferences for food quality. According to Anyasi et al. [56], the whiteness index is a measure that indicates the degree of whiteness in food items and the extent to which their color changes during the process of food preparation. Figure 4.2 presents a 3D surface plot that visually represents the influence of chemical and thermal preparation on the whiteness index. According to the findings presented in Table 4.2, the samples subjected to a 4 min blanching procedure without any chemical treatment exhibit the lowest whiteness index value. A study by Quayson et al. [57] found that the amount of color change caused by non-enzymatic processes is related to how long the blanching process lasts.

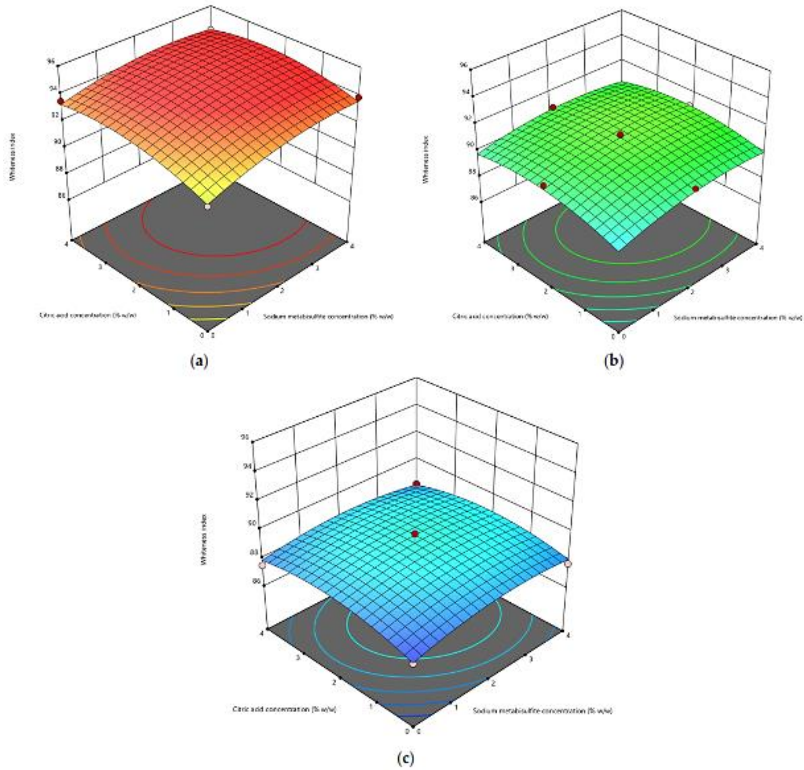

Figure 4.2. Impact of sodium metabisulfite concentration (X1) and citric acid concentration (X2) on the whiteness index (Y) of cassava slices blanched for (**a**) 0 min, (**b**) 2 min, (**c**) 4 min.

Conversely, the samples subjected to a chemical treatment including 4% citric acid and 4% sodium metabisulfite without undergoing blanching have the highest whiteness index value. Sodium metabisulfite and citric acid demonstrate notable impacts in the processing of cassava. According to a study conducted by Yongjie and Meiping [58], it was found that sulfites possess the ability to hinder both enzymatic and non-enzymatic processes. This characteristic renders them highly effective as color preservatives, especially when used in relation to perishable goods. Moreover, the utilization of metabisulfite offers supplementary advantages, such as its capacity to augment the nutritional composition of food items [59-61]. According to Ekeledo et al. [62], citric acid and similar preservatives have the ability to impede enzymatic reactions by means of oxygen removal throughout the course of food processing and storage. Application of citric acid resulted in an elevation of the whiteness index values in yam starches derived from four distinct cultivars [63].

4.3.1.3. Effective Moisture Diffusivity

Table 4.2 displays the values of effective moisture diffusivity for different pretreatment conditions. The D_{eff} values obtained for the pretreated samples exhibit a range spanning from 5.06 $\times 10^{-9}$ to 6.71 $\times 10^{-9}$ m^2/s. The findings of the study demonstrate that the application of both chemical and thermal pretreatment methods has had a significant impact on the diffusivity of moisture. Figure 4.3 shows a 3D surface plot that visually represents the influence of chemical and thermal pretreatment on effective moisture diffusivity. Additionally, it was discovered that subjecting the sample to a 2 min blanching pretreatment resulted in an increase in moisture diffusivity. Previous studies have also observed comparable outcomes in terms of the impact of pretreatment on moisture diffusivity during the drying process for blanched cassava slices [64].

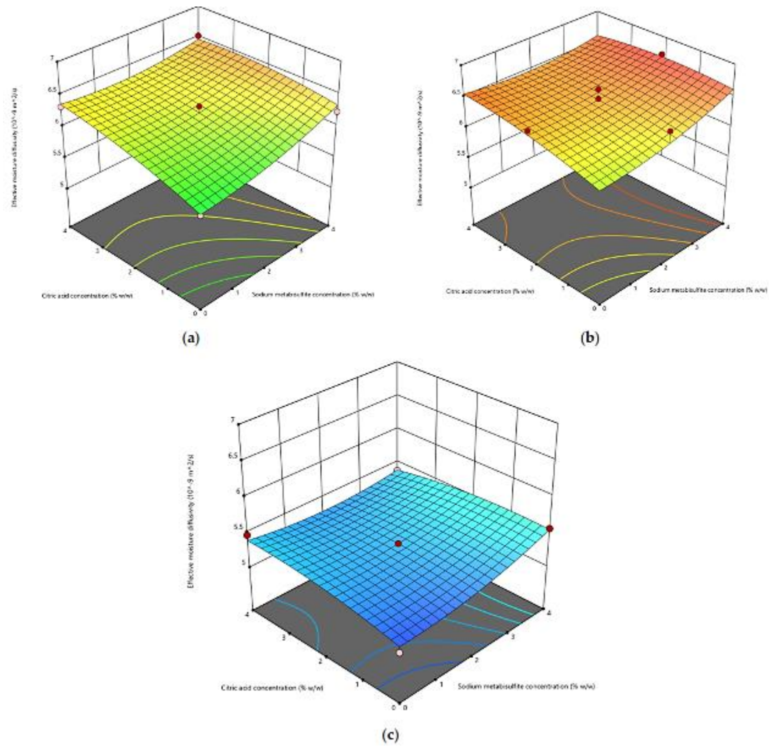

Figure 4.3. Impact of sodium metabisulfite concentration (X1) and citric acid concentration (X2) on the effective moisture diffusivity (Y) of cassava slices blanched for (a) 0 min, (b) 2 min, (c) 4 min.

It has been seen that sulfating as a pretreatment improves the process of moving water from the inside of cassava slices to the outside layers. The study conducted by Sahoo et al. [52] demonstrates that the processes of blanching and sulfiting have a substantial impact on the decrease in moisture content observed in yam slices. According to Ajala et al. [65] and

Waramit et al. [66], the D_{eff} values of cassava tubers ranged from 10^{-11} to 10^{-7} m^2/s.

4.3.1.4. Activation Energy

The importance of activation energy in the drying process is essential, as it represents the energy required by drying equipment to facilitate the diffusion of moisture from the core to the outer surface of the product [67]. According to Menshutina et al. [68], activation energy also functions as an index for estimating internal changes in the behavior of biological materials during drying. The study yielded activation energy values ranging from 29.65 to 33.28 kJ/mol, as shown in Table 4.2. Pretreatment of samples generally results in the manifestation of reduced activation energy values. This suggests that the energy demand of the drying equipment for efficient drying is reduced. Figure 4.4 depicts the influence of pretreatment on the activation energy of thin-layer dried cassava. Upon comparing Figure 4.4a–c, it becomes evident that samples exposed to shorter blanching durations exhibit a tendency towards lower activation energy levels. The application of a brief 2-min blanching treatment has been shown to yield a considerable reduction in activation energy. Similarly, the utilization of chemical treatments including sodium metabisulfite and citric acid has also been found to result in a decrease in activation energy.

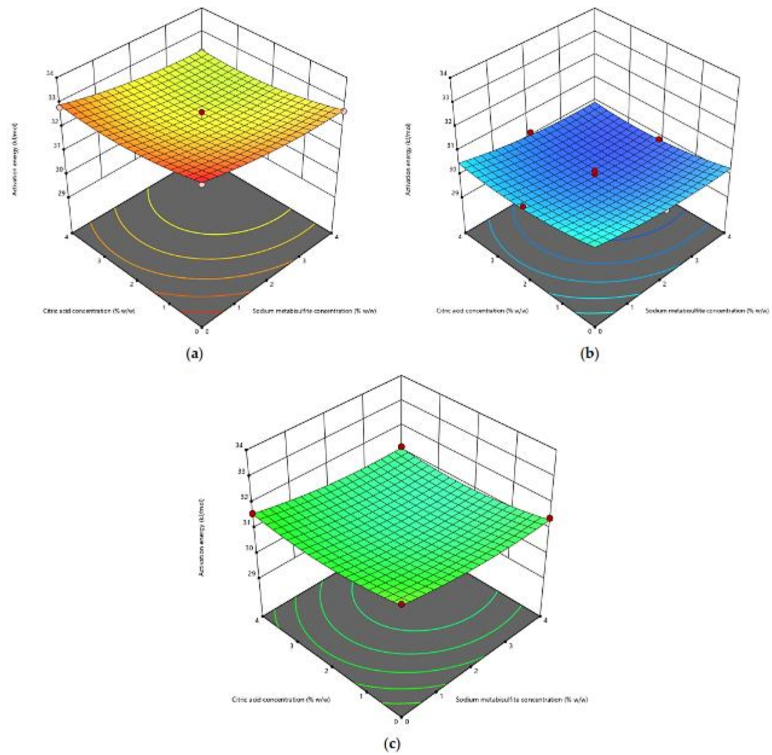

Figure 4.4. Impact of sodium metabisulfite concentration (X1) and citric acid concentration (X2) on the activation energy (Y) of cassava slices blanched for (a) 0 min, (b) 2 min, (c) 4 min.

Ajala et al. [65] reported activation energies of 30.30 kJ/mol for cassava slices, 25.18–32.46 kJ/mol for untreated and treated yam slices [69], 28.576 kJ/mol for tapioca [70], and 22.70 kJ/mol for potato slices [18]. The variability in activation energy levels seen in tubers can be attributed to various factors, including but not limited to the tuber variety, stage of maturation, size of the sample, pretreatment methods, operating conditions, and the structural composition of the tissue [52]. However, the activation energy levels observed in this investigation are consistent with the recognized range for food materials, which spans from 12.7 to 110 kJ/mol [71].

4.3.2. Modeling and Optimization of the Pretreatment Parameters

Table 4.3 displays a multiple regression analysis and analysis of variance conducted on the experimental data and the model that was generated. The associations between the dependent and independent variables were determined by using a three-level factorial quadratic regression model based on the experimental data. The ANOVA demonstrates a statistically significant regression model ($p < 0.05$), as indicated by the small p values associated with the dependent variables. The results indicate that the concentration of sodium metabisulfite, citric acid concentration, and blanching time had a significant impact ($p < 0.05$) on all tested responses.

Table 4.3. Analysis of variance (*F* value) for the response surface model of dried cassava.

Source	***Df***	**Responses**			
		MC	*WI*	D_{eff}	E_a
Model	9	45.6783 *	56.4101 *	30.7223 *	79.3221 *
SM	1	8.1370 *	12.5047 *	9.2542 *	21.1945 *
CA	1	5.4985 *	6.3233 *	5.4049 *	7.7023 *
BT	1	188.8693 *	459.5528 *	118.9614 *	118.2152 *
SM×CA	1	7.0156 *	1.2061 ^{ns}	5.0166 *	0.0403 ^{ns}
SM×BT	1	3.6824 ^{ns}	1.7137 ^{ns}	0.4045 ^{ns}	0.5067 ^{ns}
BT×CA	1	3.4778 ^{ns}	1.1678 ^{ns}	2.4842 ^{ns}	0.6746 ^{ns}
SM×SM	1	0.6323 ^{ns}	5.7665 *	1.5181 ^{ns}	2.6585 ^{ns}
CA×CA	1	5.3659 *	10.1399 *	0.8242 ^{ns}	5.3610 *
BT×BT	1	67.9459 *	10.2595 *	76.6424 *	227.1470 *

Lack of fit	5	1.07 ns	3.14 ns	1.41 ns	0.9595 ns
R^2		0.9763	0.9801	0.9651	0.9862
Adj R^2		0.9549	0.9633	0.9337	0.9738
SD		0.2616	0.4024	0.1279	0.1937
Mean		6.9595	90.8180	6.1290	31.0335
CV (%)		3.7593	0.4431	2.0862	0.6242

Note: *, significant ($p < 0.05$); ns, non-significant ($p > 0.05$); SM, sodium metabisulfite concentrations; CA, citric acid concentrations; BT, blanching time; *CV*, coefficient of variation; R^2, determination coefficient; Adj R^2, adjusted determination coefficient; *MC*, moisture content; *WI*, whiteness index; D_{eff}, effective moisture diffusivity; E_a, activation energy.

The figures presented in Figure 4.5a–d demonstrate the comparison between experimental responses and expected responses. The presented figures demonstrate the degree of linearity observed in the data, revealing a strong correlation between the experimental and anticipated values. This relationship is visually illustrated in Figure 4.5a–d. The R^2 values in Table 4.3 exhibit a range of 0.9651 to 0.9862, which suggests the statistical importance of the quadratic model. The table also indicates that all factors had a substantial impact on all replies, with a p-value less than 0.05. Hence, the updated models effectively encompass all relevant aspects [72]. The adj R^2 values, which vary between 0.9337 and 0.9738, suggest that the concentration of sodium metabisulfite, citric acid concentration, and blanching time are all statistically significant factors in the drying process of cassava slices.

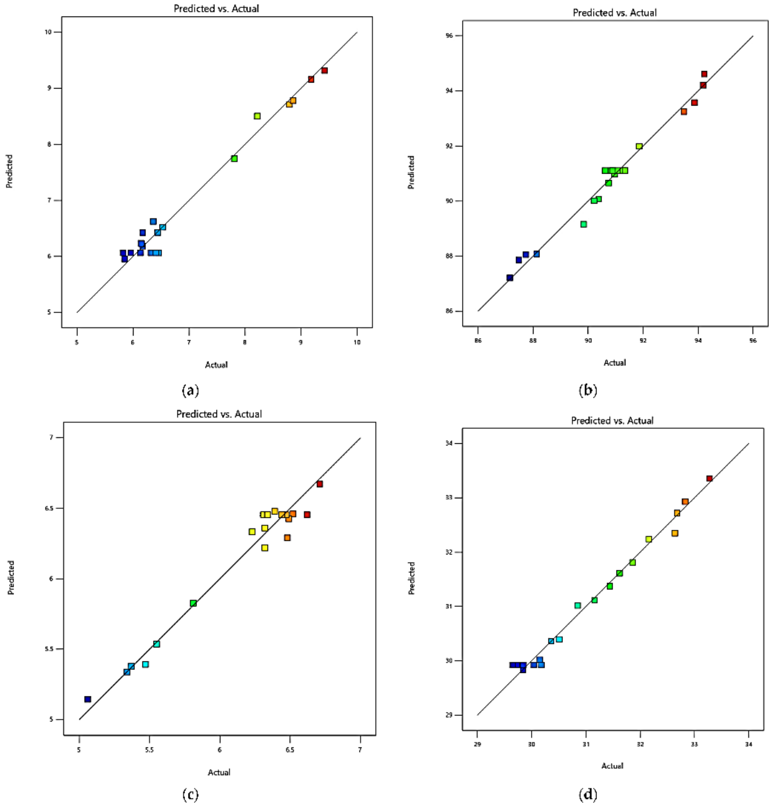

Figure 4.5. Plots comparing predicted and experimental data for responses including (a) moisture content, (b) whiteness index, (c) effective moisture diffusivity, and (d) activation energy.

The coefficient of variation is a measure that represents the ratio of the standard deviation of the estimated values to the observed mean of the dependent variable. As stated by Fasuan and Akanbi [72], the *CV* serves as an indicator of the extent to which these models may be replicated and their correctness, with a stipulated criterion of *CV* < 10%. The study demonstrates a range of results spanning from 0.44% to 3.76%, indicating that the models utilized in this research exhibit a level of reproducibility. Hence, the models developed for the evaluated reactions in dehydrated cassava slices are capable of accurately characterizing the observed process dynamics. Equations (10)–(13) encompass the mathematical formulations that serve as models for the estimation of several parameters, including moisture content (*MC*), whiteness index (*WI*), activation energy (E_a), and effective moisture diffusivity (D_{eff}), pertaining to the dehydration process of cassava slices.

$$MC = 6.06 - 0.2360(SM) - 0.1940(CA) + 1.1400(BT) + 0.1255(SM)^2 + 0.3655(CA)^2 + 1.3000(BT)^2 + 0.2450(SM)(CA) + 0.1775(SM)(BT) + 0.1725(CA)(BT) \tag{10}$$

$$WI = 91.11 - 0.4500(SM) + 0.3200(CA) - 2.7300(BT) - 0.5827(SM)^2 - 0.7727(CA)^2 + 0.7773(BT)^2 - 0.1562(SM)(CA) - 0.1863(SM)(BT) - 0.1537(CA)(BT) \tag{11}$$

$$D_{eff} = 6.45 + 0.1230(SM) + 0.0940(CA) - 0.4410(BT) + 0.0950(SM)^2 - 0.0700(CA)^2 - 0.6750(BT)^2 - 0.1013(SM)(CA) - 0.0288(SM)(BT) - 0.0713(CA)(BT) \tag{12}$$

$$E_a = 29.92 - 0.2820(SM) - 0.1700(CA) - 0.6660(BT) + 0.1905(SM)^2 + 0.2705(CA)^2 + 1.7600(BT)^2 - 0.0138(SM)(CA) + 0.0488(SM)(BT) + 0.0563(CA)(BT) \tag{13}$$

where *SM* represents the sodium metabisulfite concentration (% w/w), *CA* represents the citric acid concentration (% w/w), and BT represents the blanching time (min).

A process of optimization was conducted in order to ascertain the most favorable pretreatment conditions for the production of cassava flour, with the objective of minimizing moisture content and activation energy values while maximizing whiteness index and effective moisture diffusivity values. The investigation into the optimization of the dependent and independent variables has demonstrated that the most favorable pretreatment conditions for the drying of cassava slices are achieved through the utilization of 1.31% citric acid, 1.03% sodium metabisulfite, and a blanching duration of 1.01 min. The convergence of optimal variables yielded a moisture content of 6.19% and a whiteness index of 92.00. The activation energy for the given process was determined to be 30.98 kJ/mol, while the effective moisture diffusivity was calculated to be 6.39×10^{-9} m^2/s. Table 4.4 displays the results of a prediction and experiment that were carried out using optimized pretreatment parameters. The obtained response values suggest that the pretreatment procedure employed results in a mass transfer process that is reasonably energy-efficient and has enhanced characteristics.

Table 4.4. Results of optimized pretreatment parameters for dried cassava.

Independent Variables	Optimum Level	Predicted				Experimental				D
		MC (% db)	WI	D_{eff} (m^2/s) × 10^{-9}	E_a (kJ/mol)	MC (% db)	WI	D_{eff} (m^2/s) × 10^{-9}	E_a (kJ/mol)	
SM (% *w*/*w*)	1.03	6.19	92.00	6.39	30.98	6.23 ± 0.12	92.18 ± 0.04	6.21 ± 0.03	30.64 ± 0.25	0.74
CA (% *w*/*w*)	1.31									
BT (min)	1.01									

Note: Values reported are means ± SD. *SM*, sodium metabisulfite concentrations; *CA*, citric acid concentrations; *BT*, blanching time; *D*, desirability.

4.3.3. Microstructure of Pretreated Cassava Flour

The microscopic findings of all samples, including both untreated and treated specimens, are depicted in Figure 4.6. Uniform oval and round granules were seen in the control sample (S1) as well as in the samples subjected to chemical treatment without blanching (S2, S3, S4, and S13). On the other hand, in samples subjected to a blanching duration of 4 min, the granules displayed a range of forms and sizes. The observed phenomenon can be attributed to variations in moisture content, with lower values indicating a more loosely arranged starch polymer structure, while higher values are indicative of a more densely packed molecular structure [73]. Tacer-Caba et al. [74] reported that the application of thermal processing techniques to food materials can result in an increased level of starch gelatinization. The presence of amylose in cassava starch has a significant impact on gelatinization and retrogradation [75].

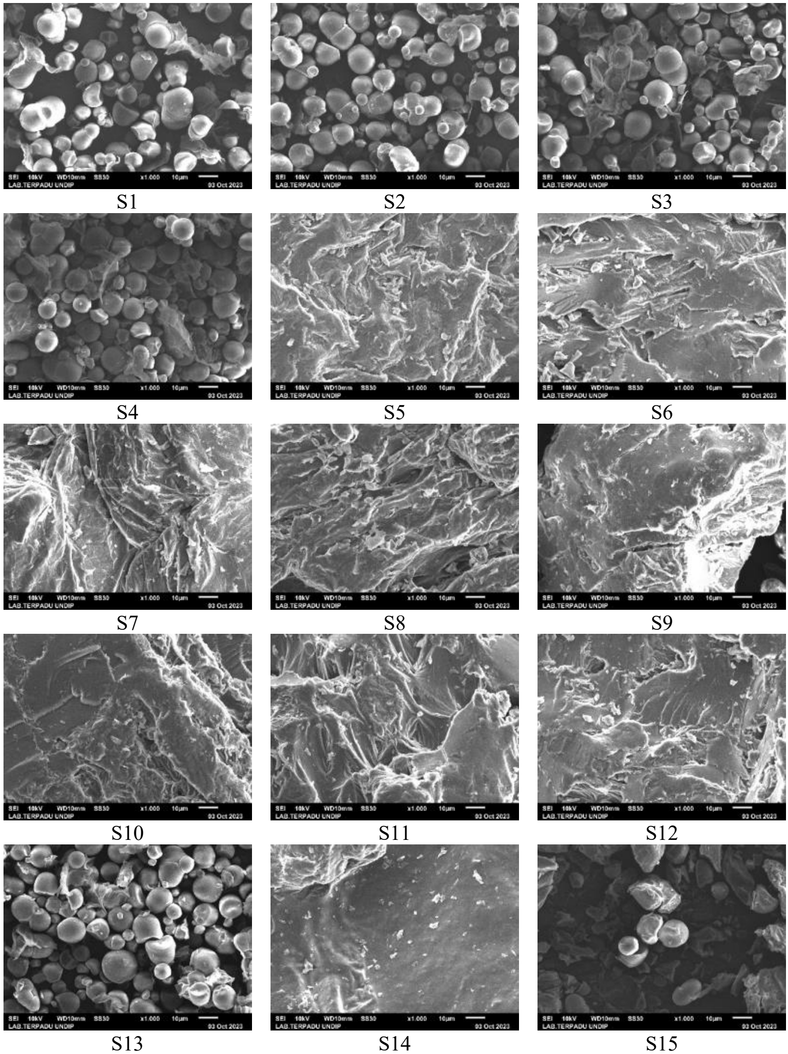

Figure 4.6. Microstructure of cassava flour with the pretreatment configuration described in Table 4.2 at 1000× magnification.

Most of the blanched samples had granules that had gelatinized, which formed large aggregates with a block-like shape and rough, void-filled surfaces. However, sample S15, which underwent treatment with a solution containing 2% sodium metabisulfite and 2% citric acid and a 2-min blanching process, showed a distinct outcome. This result deviated from the observations made in the other blanched samples. The observed pretreatment configuration exhibited granule morphology that was characterized by oval and round shapes. According to the findings of the study by Kuttigounder et al. [76], partial starch gelatinization and retrogradation had a significant impact on the morphological characteristics of granules.

4.3.4. Dehydration Kinetics of the Optimized Pretreatment Parameters

Table 4.5 displays the numerical values of model constants and statistical parameters related to the process of cassava drying. The statistical parameters show that the R^2 values range between 0.9816 and 0.9859, the χ^2 values range between 0.0351 and 0.0564, the *RMSE* values vary between 0.0015 and 0.0023, and the *SSE* values range between 0.0123 and 0.0203. According to these findings, the logarithmic model yielded the highest R^2 value and the lowest χ^2, *RMSE*, and *SSE* values. Hence, this model is the best and most appropriate for characterizing the drying characteristics of cassava slices under the indicated ideal pretreatment conditions. According to Ajala et al. [65], the logarithmic model is the most suitable for modeling the drying process of cassava slices while utilizing a tunnel dryer.

Table 4.5. Model fitting result of optimized pretreatment parameters of dried cassava.

Model	Model Parameters	R^2	χ^2	*RMSE*	*SSE*
Henderson and Pabis	$a = 0.9814$, $k = 0.2241$	0.9823	0.0564	0.0018	0.0159
Page	$k = 0.2318$, $n = 0.9914$	0.9816	0.0556	0.0021	0.0164
Logarithmic	$a = 1.0795$, $k = 0.1743$, $c = 0.1200$	0.9859	0.0351	0.0015	0.0123
Newton	$k = 0.2287$	0.9816	0.0540	0.0016	0.0164
Wang and Singh	$a = 0.1798$, $b = 0.0089$	0.9821	0.0359	0.0023	0.0203

Additional models, such as the Page model, have been employed to forecast the drying process of cassava slices using different treatments and drying techniques [76-79]. It is important to note that if different biomaterials are dried using the same pretreatment and drying configuration, it will be essential to analyze the models for each biomaterial individually in order to find the most suitable model.

4.4. Conclusions

Cassava, a tuberous plant, exhibits considerable potential for many applications in the realms of food, energy, and biofilm. The pretreatment process of cassava was modeled and optimized using a response surface methodology in order to provide efficient and cost-effective processing solutions for this plant. The results of the drying data optimization study indicate that the optimal pretreatment parameters for drying cassava slices are as follows: a citric acid concentration of 1.31%, a sodium metabisulfite concentration of 1.03%, and a blanching time of 1.01 min. The optimal conditions resulted in a moisture content of 6.19% and a whiteness index of 92.00, with an activation energy of 30.98 kJ/mol, and an effective moisture diffusivity of 6.39×10^{-9} m^2/s. The suitability of the logarithmic model was demonstrated for cassava samples that were exposed to the ideal drying conditions. The logarithmic model yielded the highest R^2 value and the lowest χ^2, *RMSE*, and *SSE* values. The results of microstructure analysis revealed that specific chemical and thermal pretreatment configurations (2% sodium metabisulfite, 2% citric acid, and blanching time for 2 min) produced particles in the form of round and oval granules. The thermal treatment of cassava tubers led to a greater extent of starch gelatinization in comparison to chemical processing. The findings disclosed in this study will have substantial implications for the advancement, evaluation, and analysis of dehydrating methodologies and systems designed for tubers.

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5. Unlocking the Potential of Solar Thermal Technology: Pretreatment-Driven Enhancement in Cassava Flour Production

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Abstract

This study explored the integration of pretreatment techniques into solar drying technology to enhance drying efficiency and product quality of cassava flour. Fresh cassava tubers were sliced and subjected to two pretreatment methods: chemical-thermal treatment (DB1; citric acid and sodium metabisulfite with blanching) and prolonged water soaking (S3D). Utilizing a solar dryer designed for optimal solar energy capture, thin-layer drying experiments revealed distinct kinetic behaviors, best modeled by the Wang and Singh equation for DB1 and the Page model for S3D samples. DB1 pretreatment significantly improved drying performance, demonstrating more efficient solar energy utilization (increased moisture diffusivity by 43% and reduced activation energy). Physicochemical analyses showed that hydrogen cyanide content was reduced by 61% (DB1) to 96% (S3D) and whiteness index increased from 89.9 to 91.4. Sensory evaluation confirmed superior texture, appearance, and overall acceptability for DB1-treated flour. The findings highlight the viability of combining targeted pretreatment methods with solar drying technology to sustainably produce high-quality cassava flour suitable for rural and energy-limited settings.

Keywords: cassava, pretreatment, solar dryer, drying kinetics, physicochemical, sensory characteristics.

5.1. Introduction

Cassava serves as a crucial staple for over 500 million people worldwide, particularly in developing nations [1]. Its resilience to various climatic conditions and capacity to flourish in suboptimal soils establish it as a dependable carbohydrate source, especially in these regions. Nonetheless, the high moisture content of fresh cassava roots, approximately 65%, renders them extremely perishable, resulting in swift post-harvest degradation [2]. This underscores the need for effective processing and preservation techniques to prolong shelf-life and uphold quality.

Drying is a commonly utilized preservation method that reduces moisture levels, therefore preventing microbial proliferation and enzymatic activity. In the case of thermally sensitive crops and food materials, drying must be executed in a controlled environment where both exposure time and drying parameters are rigorously regulated to preserve product quality and uniformity [3]. Solar drying is especially beneficial in areas with ample sunlight, providing an energy-efficient and economical alternative [4]. Solar drying leverages renewable energy from the sun, reducing dependence on fuel-based drying and making it ideal for off-grid rural applications. Thin-layer drying models are instrumental in understanding moisture removal kinetics, enabling the optimization of drying conditions. Notwithstanding the importance of these models, existing research offers limited understanding of the synergistic effects of solar drying and pretreatment techniques on cassava slices [5]. The efficacy of the drying process and the quality of the final product are profoundly affected by the drying kinetics, which delineate the rate and method of moisture extraction. Comprehending these dynamics is crucial for refining drying processes and maintaining product uniformity.

Pretreatment of cassava slices prior to drying has been shown to affect both the drying kinetics and the quality attributes of the resulting flour. Common pretreatments include blanching, soaking, and the application of chemical agents such as citric acid and sodium metabisulfite [6]. Water soaking, a more traditional method, can leach out certain undesirable compounds but also lead to nutrient loss [7]. Previous studies have explored the effects of various pretreatments on the drying behavior and quality of cassava products. For instance, the application of sodium metabisulfite has been reported to improve color retention and reduce drying time in yam flour

[8]. Blanching has been shown to enhance the rehydration capacity and textural properties of dried cassava [9]. However, comprehensive analyses comparing the combined effects of these pretreatments on drying kinetics, physicochemical properties, and sensory characteristics of cassava flour are limited.

The assessment of effective moisture diffusivity and activation energy elucidates the thermal characteristics that influence the drying process. These factors are critical for modeling and scaling up drying operations. Waramit et al. [10] demonstrated that pretreatments can modify the microstructure of cassava tissues, consequently influencing D_{eff} and E_a. The experimental analysis indicated that the drying kinetics of cassava revealed that pretreatment and thermal conditions, including hot air velocity and temperature, strongly influenced D_{eff} and E_a, with values ranging from 3.83×10^{-9} to 9.86×10^{-9} m²/s and 21.23–24.92 kJ/mol, respectively. Comparable effects were noted in research examining blanching and citric acid treatments in agricultural drying processes, which improved drying efficiency and the quality of the final product [11]. However, information regarding the impact of several pretreatment combinations, including citric acid, sodium metabisulfite, blanching, and water soaking, on the thermal properties in cassava flour manufacturing is limited.

The quality of cassava flour is significantly influenced by its physicochemical properties, which include proximate composition, pasting behavior, and color. Pretreatments alter these qualities via influencing the structure of starch granules and their interaction with other components. Blanching may induce gelatinization of starch, affecting the functional characteristics of flour. The use of chemical pretreatments such as citric acid and calcium chloride has been demonstrated to enhance the lightness and whiteness of cassava flour while altering ash and crude fiber levels [12]. Murayama et al. [13] demonstrated that pre-gelatinization considerably affects the starch content, pasting behavior, and particle size of cassava flour. Additionally, mechanical activation techniques, including ball milling, have been beneficial in altering the crystalline structure of cassava starch, hence improving its solubility and availability for enzymatic hydrolysis [14]. Nainggolan et al. [15] showed that response surface techniques indicate ideal drying conditions can produce flour with enhanced moisture content and whiteness index. The synergistic impact of chemical and thermal pretreatments on these physicochemical properties necessitates additional research to refine the processing parameters.

The sensory properties, including flavor, scent, texture, and appearance, ultimately influence customer approval of cassava flour-based goods. Solar drying, for example, has demonstrated potential in keeping desirable features like flavor and texture while maintaining nutritional content [16]. Solar drying relates to reduced microbial contamination and increased sensory characteristics when compared to sun drying [17]. Achieving a product that aligns with consumer preferences necessitates a nuanced understanding of the interplay among chemical, physical, and sensory characteristics, presenting a challenging task. Cassava flour subjected to solar drying has demonstrated enhanced customer acceptability ratings owing to superior texture and less bitter aftertaste [18]. Additionally, studies emphasize the impact of genetic and compositional features of cassava, such as starch content and carotenoids, in developing sensory preferences [19].

In light of the gaps in the existing body of knowledge, the objectives of this study are as follows: (i) to acquire thin-layer drying data and determine the most appropriate drying models for solar drying processes of treated cassava slices; (ii) to evaluate thermal attributes, such as effective moisture diffusivity and activation energy; and (iii) to investigate the influence of a variety of pretreatments on the physicochemical and sensory characteristics of cassava flour. Unlike prior studies which looked at these factors in isolation, this work provides an integrated assessment of drying kinetics and end-product quality under a solar drying context. This holistic approach is expected to yield insights for optimizing sustainable cassava processing.

5.2. Materials and Methods

5.2.1 Sample Preparation and Solar Dryer Setup

Fresh cassava tubers were obtained from local farmers at the Laguboti Market, North Sumatera. The tubers were carefully sorted to eliminate diseased or damaged samples and subsequently sliced manually to a uniform thickness of 5 ± 0.5 mm using a knife. Each experimental run consisted of a minimum of three replicates, with one kilogram of fresh cassava per replicate.

The solar dryer employed in this study was custom designed to ensure controlled drying conditions. Key specifications include:

- Dimensions: A drying chamber with dimensions of 0.5 m (L) × 0.5 m (W) × 0.5 m (H) is equipped with an attached solar collector measuring 2 m (L) × 0.5 m (W) × 0.1 m (H).
- Construction: The drying chamber was constructed with aluminum panels to maximize solar absorption. The collector is covered with transparent float glass (4 mm thick), insulated using a combination of rock wool, Styrofoam, and plywood, and the interior was coated with matte black paint to maximize heat absorption. It is oriented northward at an inclination angle of 60°.
- Airflow Design: An adjustable air inlet and an exhaust chimney were incorporated to facilitate efficient airflow and moisture removal.
- Drying Chamber: The internal dryer temperatures ranged 33-50°C during operation.

This detailed description ensures reproducibility and provides clear context for the drying kinetics observed in the experiments. The setup and design of the solar dryer used in this experiment are illustrated in Figure 5.1.

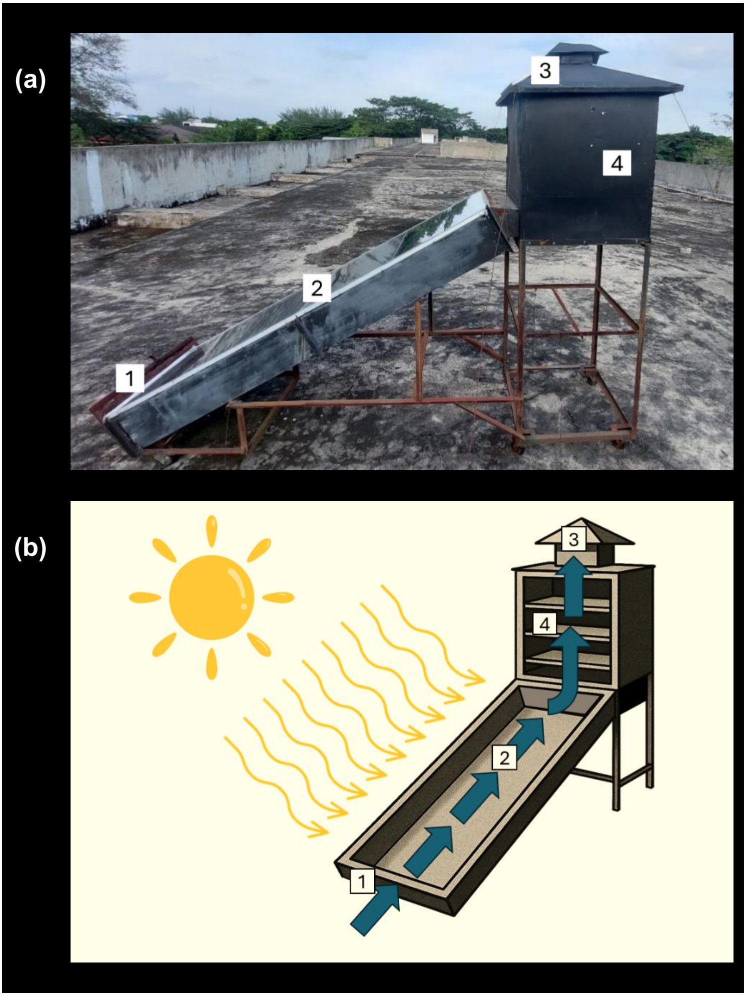

Figure 5.1. (a) Actual photograph and (b) schematic diagram of the solar dryer. Label: 1. air inlet, 2. solar collector, 3. chimney, 4. drying chamber.

5.2.2 Pretreatment and Drying Experiments

Cassava slices were subjected to a set of pretreatment regimes to determine their influence on the physicochemical attributes, drying kinetics, and sensory properties of the resulting flour. The regimes comprised combinations of citric acid, sodium metabisulfite, blanching, and water soaking. Chemical pretreatment was performed by immersing the slices in aqueous solutions of sodium metabisulfite and citric acid, whereas thermal pretreatment consisted of steam blanching at 80 °C.

One specific pretreatment method, designated as DB1, involved soaking cassava slices in a solution containing 1.31% citric acid and 1.03% sodium metabisulfite for 20 minutes, followed by steam blanching for 1 minute. Another method, labeled S3D, consisted of soaking the slices in distilled water for 72 hours at 24°C. These pretreatment configurations were based on previous studies. Untreated cassava slices, referred to as FC, served as the control group. The pretreatment configurations for DB1 and S3D were selected based on prior experimental results [15, 20] and supported by literature demonstrating that citric acid reduces browning in yam flour [21, 22] and that extended soaking effectively removes cyanogenic glycosides from cassava [23, 24]. Following these treatments, the cassava slices were drained and subjected to drying in a solar dryer. Cassava slices were dried in a solar dryer over three consecutive days (September 10–12, 2024), with all treatments conducted concurrently under standardized conditions.

Once dried, the slices were milled using a dry milling machine (HR 2115 Dry Mill Blender, PT. Philips Batam, Batam, Indonesia) and sieved through an 80-mesh sieve to achieve a uniform particle size. Flour stored at room temperature in sealed polyethylene bags and analyzed within 48 hours. Four main response variables were investigated: drying kinetics, fourier-transform infrared (FTIR) spectroscopy profiles, microstructure analysis, and proximate composition, which included measurements of moisture content, ash, crude fiber, carbohydrate, protein, fat, and caloric content. Additionally, the hydrogen cyanide (HCN) concentration, whiteness index and sensory properties of the cassava flour were also assessed.

5.2.3. Moisture Content Analysis

The determination of moisture content in the dried cassava slices was carried out using the oven drying method, following the standard procedures outlined in the Association of Official Analytical Chemists (AOAC) [25] guidelines. This method involved drying the samples at a controlled temperature until a constant weight was achieved. The moisture content was calculated on a dry weight basis using Equation (1).

MC represents the moisture content expressed as a percentage,

$$MC\ (\%) = \frac{W_t - W_i}{W_i} \times 100 \quad (1)$$

W_t is the weight of the sample at a given time *t* during the drying process, and W_i is the weight of the sample after drying to a constant weight. The calculated moisture content provided an indication of the water retained in the cassava slices after the drying process, which is a critical parameter for assessing the effectiveness of the drying method and the quality of the dried product.

5.2.4 Drying Kinetics

Following the application of the specified pretreatment conditions, drying experiments were conducted to assess the drying behavior of cassava slices. To quantify the drying process, the moisture ratio (*MR*) was calculated based on the simplified Fick's diffusion law, as represented in Equation (2).

$$MR = \frac{M}{M_o} \quad (2)$$

M represents the moisture content of cassava at any given time during the drying process, and M_o denotes the initial moisture content. The experimental data obtained were statistically analyzed using five widely established thin-layer drying models, selected due to their frequent application in root-crop drying studies [26–28], as summarized in Table 5.1.

Table 5.1. Mathematical drying models applied to cassava drying.

Model's name	Model	References
Henderson and Pabis	$MR = a \exp(-kt)$	[29]
Page	$MR = \exp(-kt^n)$	[30]
Logarithmic	$MR = a \exp(-kt) + c$	[31]
Newton	$MR = \exp(-kt)$	[32]
Wang and Singh	$MR = 1 + at + bt^2$	[33]

The performance of the drying models was evaluated using statistical metrics, including the coefficient of determination (R^2), sum of squares error (*SSE*), reduced chi-square ($\chi^2_{pred,i}$), and root mean square error (*RMSE*), calculated using the following equations:

$$R^2 = \frac{1 - [\sum_{i=1}^{n}(MR_{pre,i} - MR_{exp,i})^2]}{[\sum_{i=1}^{n}(MR_{pre,i} - MR_{exp,i})^2]} \quad (3)$$

$$SSE = \frac{\sum_{i=1}^{n}(MR_{pre,i} - MR_{exp,i})^2}{N} \quad (4)$$

$$\chi^2 = \frac{\sum_{i=1}^{n}(MR_{pre,i} - MR_{exp,i})^2}{N - z} \quad (5)$$

$$RMSE = \left[\frac{\sum_{i=1}^{n}(MR_{pre,i} - MR_{exp,i})^2}{N}\right]^{\frac{1}{2}} \quad (6)$$

N denotes the number of experimental observations, $MR_{pred,I}$ represents the predicted moisture ratio values, $MR_{exp,i}$ refers to the experimentally determined moisture ratio values, and z corresponds to the number of parameters in the drying model. A higher R^2 value, along with lower values of *SSE*, $\chi^2_{pred,i}$, and *RMSE*, indicates a better fit of the drying model to the experimental data.

5.2.5 Determination of the Effective Moisture Diffusivity

The effective moisture diffusivity (D_{eff}) of cassava slices was determined based on Fick's second law of diffusion, considering slab geometry [34]. The calculation assumed a uniform initial moisture content, negligible external mass transfer resistance, constant diffusivity, and insignificant shrinkage, as represented in Equation (7):

$$MR = \frac{M_i - M_e}{M_o - M_e} = \frac{8}{\pi^2}\sum_{n-1}^{\infty}\frac{1}{(2n+1)^2} exp\left(\frac{-(2n+1) \times \pi^2 \times D_{eff} \times t}{4H^2}\right) \quad (7)$$

where MR denotes the dimensionless moisture ratio, M_i is the moisture content at i time (% db), M_e is the equilibrium moisture content (% db), M_o the initial moisture content (% db). The variable H represents half the thickness of the cassava slices (m), t is the drying time (min), and n is a positive integer.

For practical analysis, Equation (7) was transformed into a logarithmic form to simplify the estimation of D_{eff}, as shown in Equation (8):

$$Ln(MR) = Ln\left(\frac{8}{\pi^2}\right) - \left(\frac{\pi^2 \times D_{eff} \times t}{4H^2}\right) \quad (8)$$

This transformation allows for a linear representation of the relationship described by Fick's law. By plotting *Ln (MR)* against drying time (t), a straight-line graph is obtained. The slope of this graph corresponds to $\frac{\pi^2 \times D_{eff}}{4H^2}$, enabling the calculation of the effective moisture diffusivity.

5.2.6 Determination of the Activation Energy

Equation (9) expresses the calculation of activation energy, where the relationship between effective moisture diffusivity and temperature is typically characterized by the Arrhenius equation [35].

$$D_{eff} = D_o exp\left(-\frac{E_a}{RT_a}\right) \quad (9)$$

where E_a represents the activation energy (kJ/mol), R represents the universal gas constant (8.3143 J/mol.K), T_a represents the absolute air temperature (K) and D_o represents the pre-exponential factor from the Arrhenius equation (m^2/s).

5.2.7 FTIR Analysis

FTIR spectroscopy was performed using a Perkin-Elmer UATR Spectrum Two (Perkin Elmer, Rodgau, Germany) to analyze the molecular and structural composition of the cassava flour samples. This technique provides detailed insights into the functional groups and chemical bonds present in the samples, enabling a comprehensive understanding of their chemical structure. All cassava flour samples were subjected to this analysis to ensure consistency and reliability in the identification of their compositional characteristics.

5.2.8 Microstructure Analysis

The morphology of cassava flour samples was analyzed using a scanning electron microscope (SEM) (Model JSM-6510LA, Jeol Ltd., Tokyo, Japan). This analysis aimed to evaluate the effects of various pretreatments on the particle morphology and structural characteristics of the cassava flour. The SEM was operated at an accelerating voltage of 10 kV to ensure optimal image resolution and clarity. High-resolution micrographs of the samples were captured, allowing for a detailed examination of surface features, particle size, and textural attributes influenced by the pretreatment methods.

5.2.9 Proximate Composition and HCN Concentration Analysis

The proximate composition of cassava flour, including moisture content, ash, crude fiber, carbohydrate, total protein, fat, and calorie content, as well as the concentration of hydrogen cyanide (HCN), were analyzed using the standard procedures outlined by the AOAC [25]. These methods ensure accurate and reliable measurements of the nutritional and chemical properties of the cassava flour, providing a comprehensive profile of its compositional attributes.

5.2.10 Color Measurement

The color properties of cassava flour samples were analyzed in this study using a precision colorimeter (model CS-10, manufactured by Hangzhou Caipu Technology Co., Ltd., Hangzhou, China). Prior to the measurements, the instrument was calibrated against standard white and black reference tiles to ensure accuracy and consistency in the readings. The evaluated color parameters included L*, a*, and b*. The L* parameter represents lightness, with values ranging from 0 (black) to 100 (white). The a* parameter quantifies the red-green spectrum, where positive values signify red and negative values indicate green. Similarly, the b* parameter measures the yellow-blue spectrum, with positive values denoting yellow and negative values indicating blue. To further quantify the color characteristics of cassava flour, the whiteness index (*WI*) was calculated. This index integrates the L*, a*, and b* values into a single measure, providing an assessment of the sample's whiteness. The calculation followed the formula as shown below:

$$WI = 100 - \sqrt{a^{*2} + b^{*2} + (100 - L^{*})^{2}} \tag{10}$$

5.2.11 Determination Sensory Characteristics

To evaluate the sensory characteristics of the cassava flour, 40 untrained assessors were recruited, consisting of an equal gender distribution (20 males and 20 females). The participants were selected from the Department of Bioprocess Engineering, Institut Teknologi Del. These assessors were tasked with evaluating key quality attributes of the cassava flour, including texture, taste, appearance, color, and overall acceptability. Each participant evaluated coded samples in three replicates to ensure reliable results. The assessments were conducted using a 9-point hedonic scale, designed in accordance with ISO 8586-1 sensory analysis guidelines. The scale ranged from 1 to 9, where 1 represented "dislike extremely," 2 "dislike very much," 3 "dislike slightly," 4 "dislike," 5 "neither like nor dislike," 6 "like," 7 "like slightly," 8 "like very much," and 9 "like extremely." This structured evaluation method ensured a comprehensive and standardized assessment of the sensory properties.

5.2.12 Data Analysis

One-way analysis of variance (ANOVA), followed by Tukey's post hoc test at a significance level of $p < 0.05$, and moisture ratio analyses were conducted using Minitab® version 19.1 (Minitab LLC, State College, PA, USA). Nonlinear regression techniques were applied to assess the parameters of the selected drying models. The evaluation of the models' performance was based on several statistical criteria. Specifically, a higher coefficient of determination (R^2) indicated a better fit of the model to the observed data. Additionally, lower values of the sum of squared errors (SSE), reduced chi-square ($\chi^2_{pred,i}$), and root mean square error ($RMSE$) were considered as indicators of improved model accuracy and reliability. These statistical measures collectively ensured a robust and precise evaluation of the drying model parameters. Furthermore, Principal Component Analysis (PCA) and correlation heat map analysis were performed using Python to explore underlying data patterns and relationships.

5.3. Results and Discussion

5.3.1. Drying Kinetics

This study evaluates the numerical thin-layer drying models applied to cassava dried using solar dryers. The results are summarized in Table 5.2, showing distinct performance metrics for different models and sample types. For the FC sample, the R^2 ranged from 0.9403 to 0.9848, with all drying models achieving R^2 values above 0.90. The χ^2 values ranged from 0.0223 to 0.8435, RMSE from 0.0011 to 0.1896, and *SSE* from 0.99 to 1.5169. Among the models tested, the Wang and Singh model demonstrated the highest accuracy for the FC sample, as evidenced by the highest R^2 value (0.9848) and the lowest χ^2 (0.0223), *RMSE* (0.0011), and *SSE* (0.0099).

For the S3D sample, the R^2 values ranged from 0.9420 to 0.9973, with all models maintaining R^2 values above 0.90. The χ^2 values ranged from 0.0042 to 0.3845, *RMSE* values from 0.0003 to 0.0610, and *SSE* values from 0.0027 to 0.4880. The Page model emerged as the most suitable for the S3D sample, achieving the highest R^2 value (0.9973) and the lowest χ^2 (0.0042), *RMSE* (0.0003), and *SSE* (0.0027). For the DB1 sample, the R^2 values ranged from 0.9348 to 0.9837, χ^2 values from 0.0215 to 0.5147, *RMSE* from 0.0014 to 0.0959, and *SSE* from 0.0128 to 0.7673. The Wang and Singh model exhibited the highest predictive accuracy for the DB1 sample, marked by the highest R^2 and the lowest χ^2, *RMSE*, and *SSE* values.

Table 5.2. Model fitting results for the solar drying process of pretreated cassava slices.

Model	Model parameters	R^2	χ^2	*RMSE*	*SSE*
Sample FC					
Henderson and Pabis	a = 1.0415, k = 0.1116	0.9403	0.0919	0.0041	0.0370
Page	k = 0.0502, n = 1.3891	0.9645	0.0549	0.0027	0.0223
Logarithmic	a = 17.4699, k = 0.0004, c = 16.4961	0.9810	0.8435	0.1896	1.5169
Newton	k = 0.1045	0.9439	0.0994	0.0041	0.0408
Wang and Singh	a = 0.0657, b = 0.0010	**0.9848**	**0.0223**	**0.0011**	**0.0099**
Sample S3D					
Henderson and Pabis	a = 1.0944, k = 0.1391	0.9420	0.1144	0.0057	0.0520
Page	k = 0.0317, n = 1.7342	**0.9973**	**0.0042**	**0.0003**	**0.0027**
Logarithmic	a = 118.923, k = 0.0005, c = 117.833	0.9939	0.3845	0.0610	0.4880

Newton	*k* = 0.1230	0.9515	0.1521	0.0070	0.0708
Wang and Singh	*a* = 0.0695, *b* = 0.0017	0.9949	0.0140	0.0005	0.0046
Sample DB1					
Henderson and Pabis	*a* = 1.0657, *k* = 0.1358	0.9348	0.1135	0.0059	0.0538
Page	*k* = 0.0405, *n* = 1.6074	0.9812	0.0220	0.0020	0.0165
Logarithmic	*a* = 140.103, *k* = 0.0003, *c* = 139.031	0.9827	0.5147	0.0959	0.7673
Newton	*k* = 0.1244	0.9412	0.1359	0.0062	0.0628
Wang and Singh	*a* = 0.0763, b = 0.0008	**0.9839**	**0.0215**	**0.0014**	**0.0128**

The results indicate that the Page and Wang and Singh models are the most effective for describing the drying kinetics of cassava flour under non-linear analysis. The Page model is best suited for the S3D sample, while the Wang and Singh model is more applicable to the FC and DB1 samples. These results align with the conclusions of Suherman et al. [36], that identified the Page model as optimal for modeling the drying process of cassava slices using hybrid solar dryers. Additionally, alternative models including the Henderson and Pabis model [37], Logarithmic model [38], and Two-Term model [39] have been employed to forecast cassava drying kinetics under diverse treatment conditions and solar drying setups. It is imperative to underscore that for biomaterials undergoing various pretreatments and drying configurations, a distinct evaluation of drying models is necessary to choose the most appropriate one for each specific process. This work highlights the significance of model selection to optimize drying efficiency and improve the comprehensive understanding of the drying kinetics of pretreated cassava.

5.3.2. Thermal Properties

Table 5.3 presents effective moisture diffusivity and activation energy for cassava slices under various pretreatments during solar drying. The D_{eff} values increased significantly following pretreatments, specifically by 36.7% for S3D (5.07×10^{-9} m²/s) and 43.1% for DB1 (5.31×10^{-9} m²/s), compared to the control (FC, 3.71×10^{-9} m²/s). These enhancements suggest that chemical pretreatments involving soaking and blanching effectively improve the moisture diffusion rate by softening cellular structures and increasing permeability, thus facilitating rapid moisture migration during drying [40].

Table 5.3. Thermal properties for solar drying of cassava slices.

Pretreatment	D_{eff} (m²/s) × 10^{-9}	E_a (kJ/mol)
FC	3.71 ± 0.18^b	33.23 ± 0.35^a
S3D	5.07 ± 0.24^a	30.42 ± 0.63^b
DB1	5.31 ± 0.02^a	30.70 ± 0.51^b

Note: Values are mean ± SD (n = 3); values sharing the same superscript letter within a column are not significantly different ($p < 0.05$).

Activation energy values significantly decreased after pretreatments. Specifically, the E_a for S3D (30.42 kJ/mol) and DB1 (30.70 kJ/mol) were lower than the control (33.23 kJ/mol). Lower E_a values indicate reduced energy requirements for initiating moisture removal, signifying improved drying efficiency [41]. Pretreatments involving chemical soaking or blanching soften tissue matrices, reduce cellular rigidity, and enhance water mobility, promoting faster drying rates [12].

The DB1 sample demonstrated an optimal combination of high D_{eff} and low E_a values, making it highly suitable for industrial applications. This treatment accelerates the drying process, saves time, and maintains product quality [42]. Mudafale et al. [43] indicated that the integration of solar drying with chemical treatments significantly improves drying efficiency, leveraging renewable energy sources and promoting energy sustainability. Chemical treatments such as blanching with citric acid and sodium metabisulfite (DB1) and soaking (S3D) significantly improve D_{eff} and reduce E_a. These improvements facilitate mass transfer and enhance drying efficiency in cassava, offering promising applications in the food processing industry.

5.3.3. FTIR Analysis

Figure 5.2 illustrates the FTIR analysis of pretreatment dried cassava slices, intended to examine the crystalline structure and vibrational modes of chemical bonding. The FC spectrum illustrates the inherent properties of unprocessed cassava starch. The hydroxyl group (O-H) at around 3286 cm^{-1} has reduced intensity relative to the other samples, signifying a diminished hydration level. The carbonyl band (C=O) at 1625.50 cm^{-1} verifies the existence of native starch absent of extraneous chemical interactions.

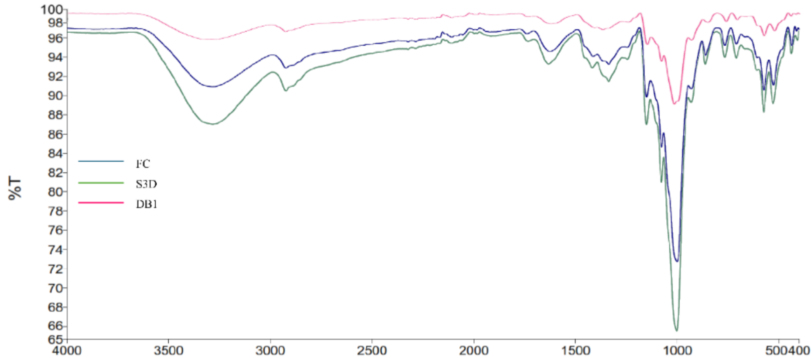

Figure 5.2. FTIR spectra of solar-dried cassava flour.

The hydroxyl group (O-H) in the DB1 spectrum at 3289.47 cm^{-1} exhibits increased intensity (96.22%T), indicating augmented interaction with water as a result of chemical treatment. The carbonyl band (C=O) moves to 1619.32 cm^{-1} with enhanced intensity (98.09%T), signifying interactions between starch and citric acid. Ačkar et al. [44] indicated that chemical treatment using organic acids improves the solubility and hydration characteristics of starch. The heightened intensity of the C-O band at around 1148.50 cm^{-1} indicates structural alterations in carbohydrates due to chemical reactions. The S3D spectrum indicates a hydroxyl group (O-H) at 3284.67 cm^{-1} with diminished intensity (87.04%T), ascribed to deterioration during the soaking process. The carbonyl band (C=O) shifts to 1631.62 cm^{-1} with diminished intensity (93.31%T), signifying structural alterations in starch resulting from enzymatic or microbial activity [45]. A reduction in the intensity of the C-O band at 1149.59 cm^{-1} indicates partial breakdown of carbohydrates.

The DB1 sample exhibits an augmentation in the intensity of the carbonyl and hydroxyl bands, signifying an alteration in starch structure. Interactions between starch and citric acid or sodium metabisulfite may lead to esterification or other derivatives that improve solubility and hydration. The shift of the C=O band to lower wavenumber in DB1 suggests esterification between citric acid and hydroxyl groups of amyloses, improving solubility [46]. Increased O–H intensity correlates with enhanced hydrogen bonding networks, which may underlie improved hydration capacity. In contrast, the S3D sample demonstrates a reduced hydration impact relative to DB1, with indications of carbohydrate breakdown resulting from enzymatic or microbial activity during soaking. The FC sample exhibits diminished intensity in hydroxyl and carbonyl bands, indicating the intrinsic characteristics of cassava starch absent exogenous modifications [47].

5.3.4. SEM Analysis

Figures 5.3a, 5.3b, and 5.3c present SEM images of cassava flour dried using a solar dryer at 1000x magnification. In the FC sample, the structure exhibits substantial variability, predominantly featuring spherical particles. The absence of prior treatments allows the microstructure to retain the natural characteristics of cassava, including the presence of amylose and amylopectin in their native forms. The observed rough surfaces indicate the lack of intensive washing processes to remove non-starch components.

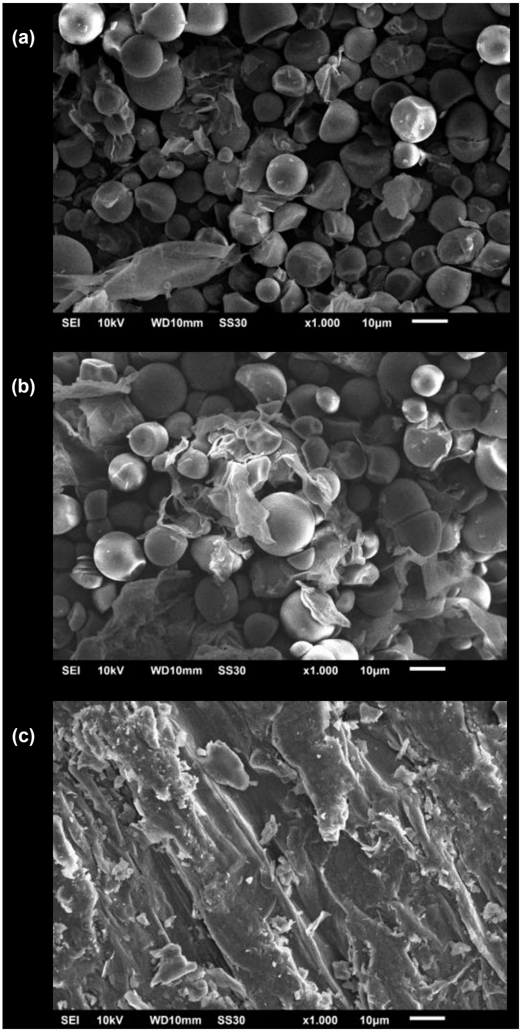

Figure 5.3. Microstructure of solar-dried cassava with pretreatment observed at 1000X magnification: (a) fresh cassava (FC); (b) pretreated with S3D; (c) pretreated with DB1.

In the S3D sample, soaking for 72 hours significantly influences the microstructure. Spherical particles remain visible, but the surface appears smoother compared to the FC sample. This treatment facilitates the dissolution of water-soluble components such as proteins and other compounds, contributing to impurity reduction and creating a more homogeneous texture [16]. In the DB1 sample, the microstructure undergoes significant alterations, characterized by large block-like and irregular masses, along with voids and rough surfaces. Chemical treatments such as blanching promote starch gelatinization, resulting in a denser, more uniform microstructural arrangement. DB1's block-like granules result from partial gelatinization during blanching, as observed by Dudu et al. [48], which enhances water absorption but reduces porosity relative to S3D.

These microstructural differences illustrate how treatment methods affect the functional properties of cassava flour. Treatments such as DB1 are suitable for enhancing stability and improving storage characteristics, while S3D offers advantages for applications requiring high porosity, such as ingredients in instant food formulations [49]. Dehnad et al. [50] reported that solar drying or blanching combined with humidity control, can improve the physical and functional properties of cassava flour, potentially expanding its applications in the food industry.

5.3.5. Physicochemical Properties

Table 5.4 summarizes the proximate composition, hydrogen cyanide (HCN) concentration, and whiteness index of solar-dried cassava flour from fresh cassava (FC), 72 h water-soaked (S3D), and chemical-thermal-blanched (DB1) samples. Moisture content was significantly reduced by both pretreatments (S3D: 11.51 ± 0.18 %; DB1: 11.66 ± 0.17 %) compared to the control (FC: 13.82 ± 0.29 %) ($p < 0.05$), indicating that soaking and citric-acid/sodium-metabisulfite blanching facilitate water removal during solar drying. Chaethong and Pongsawatmanit [51] demonstrated that the application of sodium metabisulfite and citric acid affects the moisture content and quality of the final product.

Table 5.4. Physicochemical properties of solar-dried cassava flour.

Parameter	FC	S3D	DB1
Proximate			
Moisture (%)	13.82 ± 0.29^{a}	11.51 ± 0.18^{b}	11.66 ± 0.17^{b}
Ash (%)	2.15 ± 0.32^{b}	1.40 ± 0.23^{c}	2.95 ± 0.33^{a}
Crude Fiber (%)	1.67 ± 0.13^{a}	1.55 ± 0.13^{a}	1.87 ± 0.15^{a}
Carbohydrate (%)	86.83 ± 0.82^{a}	83.47 ± 0.68^{b}	85.99 ± 0.89^{a}
Total Protein (%)	3.41 ± 0.26^{a}	3.22 ± 0.32^{a}	3.29 ± 0.14^{a}
Fat (%)	0.61 ± 0.07^{a}	0.53 ± 0.05^{a}	0.48 ± 0.04^{a}
Calorie (Kcal/100g)	361.67 ± 0.57^{a}	348.24 ± 0.60^{c}	357.86 ± 0.65^{b}
HCN concentration			
HCN (ppm)	32.16 ± 0.43^{a}	1.26 ± 0.11^{c}	12.64 ± 0.58^{b}
Color			
Whiteness Index	89.92 ± 0.42^{b}	91.43 ± 0.50^{a}	91.36 ± 0.52^{a}

Note: Values are mean ± SD (n = 3); values sharing the same superscript letter within a row are not significantly different ($p < 0.05$).

Ash content increased in DB1 (2.95 ± 0.33 %) relative to FC (2.15 ± 0.32 %) and S3D (1.40 ± 0.23 %) ($p < 0.05$), reflecting mineral retention or uptake from the chemical pretreatment. Deng et al. [52] reported that chemical pretreatment prior to drying can affect the mineral content and other volatile compounds in food products. In contrast, crude fiber (1.55–1.87 %), total protein (3.22–3.41 %), and fat (0.48–0.61 %) did not differ significantly among treatments ($p > 0.05$), suggesting these macronutrients are largely unaffected by soaking or blanching. Carbohydrate content was highest in FC (86.83 ± 0.82 %) and DB1 (85.99 ± 0.89 %) and significantly lower in S3D (83.47 ± 0.68 %) ($p < 0.05$), consistent with the loss of soluble solids during prolonged soaking. Accordingly, the energy value of S3D (348.24 ± 0.60 kcal/100 g) was lower than that of DB1 (357.86 ± 0.65 kcal/100 g) and FC (361.67 ± 0.57 kcal/100 g). HCN concentration decreased markedly from 32.16 ± 0.43 ppm in FC to 12.64 ± 0.58 ppm in DB1 and 1.26 ± 0.11 ppm in S3D ($p < 0.05$), demonstrating the efficacy of 72 h soaking in leaching cyanogenic glycosides, as confirmed in studies on the efficiency of toxin removal using solar energy-based drying methods [53].

Both pretreatments produced a significant increase in whiteness index (S3D: 91.43 ± 0.50; DB1: 91.36 ± 0.52) compared to FC (89.92 ± 0.42) ($p < 0.05$), indicating improved visual quality likely due to pigment removal and minimized non-enzymatic browning [54]. Combining solar drying with targeted pretreatments optimizes moisture reduction, minimizes HCN content, and enhances mineral and visual quality of cassava flour, while preserving fibre, protein, and fat, supporting a safe, nutritious product suitable for food applications [55].

5.3.6. Sensory Characteristics

Table 5.5 summarizes the sensory evaluation results of solar-dried cassava flour samples (DB1, S3D, and FC) assessed by untrained panelists (n = 3 per sample). Overall, DB1 treatment consistently received the highest scores across all attributes, indicating superior sensory quality. In terms of texture, DB1 and S3D achieved significantly higher scores (8.63 and 8.38, respectively) than the untreated control (FC, 7.45), suggesting that pretreatments involving soaking or chemical-thermal methods enhance flour texture. This agrees with previous findings that pretreatments, particularly the application of sodium metabisulfite and citric acid, can improve the softness and cohesiveness of cassava flour [56].

Table 5.5 Sensory characteristics of solar-dried cassava flour.

Sample	Attribute				
	Texture	**Taste**	**Appearance**	**Colour**	**General Acceptability**
DB1	8.63 ± 0.48^{a}	8.60 ± 0.49^{a}	8.73 ± 0.44^{a}	8.55 ± 0.49^{a}	8.63 ± 0.48^{a}
S3D	8.38 ± 0.49^{a}	8.55 ± 0.50^{a}	8.48 ± 0.52^{b}	8.65 ± 0.48^{a}	8.50 ± 0.50^{a}
FC	7.45 ± 0.52^{b}	8.53 ± 0.50^{a}	8.15 ± 0.62^{c}	7.95 ± 0.62^{b}	8.03 ± 0.57^{b}

Note: Values are mean ± SD; values sharing the same superscript letter within a column are not significantly different ($p < 0.05$).

For taste, all samples received similarly high ratings (8.5), with no significant differences observed among treatments. Although FC was slightly lower, it was statistically comparable to S3D and DB1, indicating that pretreatment had a minimal influence on perceived taste under the conditions tested. Regarding appearance, DB1 again scored the highest (8.73), followed by S3D (8.48) and FC (8.15), with each sample being significantly different. The enhancement observed in the DB1 and S3D samples may be due to reduced browning and better retention of color, as soaking and blanching are known to inhibit enzymatic reactions and improve surface brightness [12]. In the colour category, both DB1 and S3D outperformed FC significantly. S3D showed the highest colour score (8.65), followed by DB1 (8.55), while FC lagged behind (7.95). This supports earlier reports that prolonged soaking in distilled water reduces enzymatic browning, resulting in a lighter and more appealing flour color [15].

For general acceptability, DB1 exhibited the highest overall score (8.63), significantly surpassing FC (8.03), while S3D (8.50) remained statistically similar to DB1. The combination of chemical and thermal pretreatments in DB1 contributed to this enhanced acceptability by improving texture, appearance, and color simultaneously [57]. The untreated FC sample consistently showed the lowest ratings across most sensory parameters, emphasizing the importance of pretreatment. Notably, the 72-hour soaking in S3D improved the flour's appearance and color through microbial fermentation, although it was less effective than DB1 in enhancing general acceptability. Based on the panelists’ evaluations, the DB1 pretreatment was the most effective approach to improving the sensory characteristics of solar-dried cassava flour.

5.3.7. Comparative assessment of various pretreatment methods.

Figure 5.4a presents a PCA biplot that distinctly delineates the physicochemical variations induced by different cassava pretreatment methods. PCA is a well-established multivariate technique in drying studies, employed to reduce dimensionality by summarizing interrelated physicochemical variables into a smaller set of uncorrelated components [58, 59]. The first principal component (PC1) accounted for 71.9% of the total variance, while the second principal component (PC2) explained an additional 28.1%, collectively representing the entire variability in the dataset. The PCA scatterplot identifies three distinct clusters corresponding to the different pretreatments. Untreated cassava flour is isolated on the positive side of PC1, characterized by high moisture, activation energy, fat, total protein, calorie, carbohydrate, and cyanide levels. Conversely, DB1 treatment is distinctly separated on the positive side of PC2, closely associated with higher crude fiber and ash contents. The S3D treatment is positioned negatively on PC1, prominently linked to higher whiteness index values and effective moisture diffusivity, indicative of improved drying performance and visual quality.

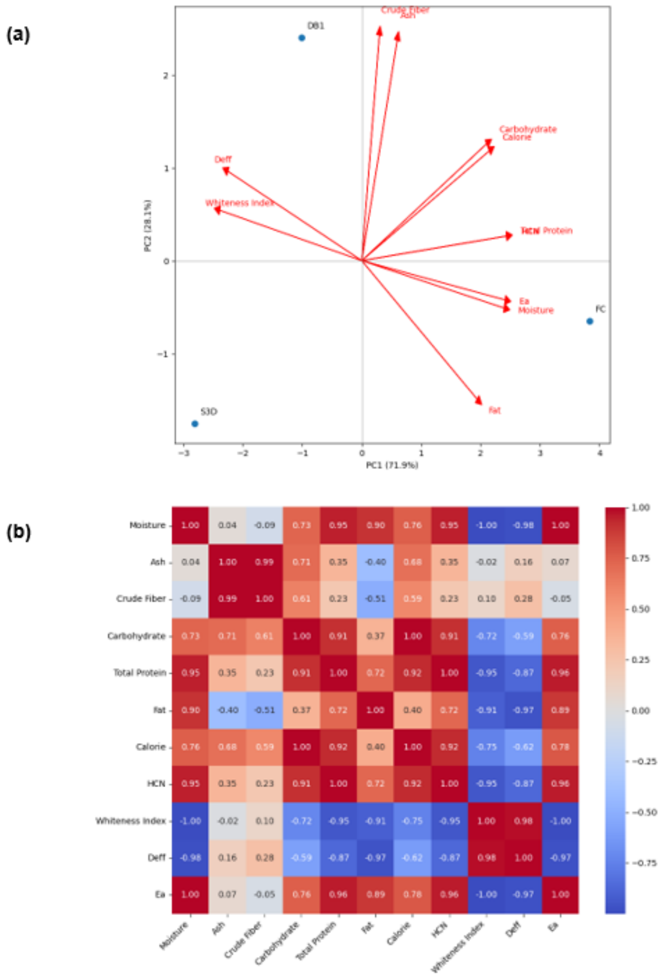

Figure 5.4. Comparative analysis of pretreatment effects: (a) PCA biplot of physicochemical and drying parameters (showing clustering of samples FC, S3D, DB1); (b) Pearson correlation heatmap among key quality and drying parameters.

The correlation heatmap (Figure 5.4b) further reinforces the PCA results by identifying strong associations among physicochemical parameters. A notably positive correlation was observed between moisture, HCN, total protein, calorie, and carbohydrate, suggesting these components co-vary significantly. Additionally, ash content demonstrated a robust positive correlation with crude fiber content, suggesting that mineral-rich and fibrous components are similarly retained in specific pretreatments, particularly DB1. Conversely, whiteness index was negatively correlated with moisture, fat, and HCN, reflecting that samples with lower moisture and cyanide levels typically exhibit a brighter appearance. Moreover, D_{eff} exhibited strong negative correlations with moisture and HCN, underscoring that increased drying efficiency is associated with reduced moisture and detoxification effectiveness. These comprehensive insights are critical for selecting appropriate pretreatment methods to optimize cassava flour quality and safety for drying applications and starch modification processes.

5.4. Conclusion

This study systematically evaluated the effects of water soaking and combined chemical-thermal pretreatment on thin-layer solar drying of cassava slices. The results demonstrate that integrating citric acid and sodium metabisulfite soaking with brief blanching (the DB1 pretreatment) markedly enhances drying performance and final flour quality. The Page model provided the best fit for S3D kinetics ($R^2 = 0.9973$), while the Wang and Singh model was most appropriate for both untreated and DB1 samples ($R^2 \geq 0.9839$). Compared to the control, the DB1 treatment increased effective moisture diffusivity by

43.1% and reduced activation energy to 30.7 kJ/mol, translating into shorter drying times and lower energy requirements. Physicochemical analyses revealed that pretreatments lowered HCN content by up to 61% in the chemical-treated sample, improved whiteness index, and enhanced mineral retention without compromising macronutrient composition. SEM and FTIR data confirmed structural modifications conducive to higher hydration and solubility. Sensory evaluation by untrained panellists rated DB1-treated flour highest for texture, appearance, and overall acceptability, while S3D also yielded significant gains in brightness and texture.

These findings underscore the value of combining chemical and thermal pretreatments with solar-thermal technology for decentralized cassava flour production. The DB1 protocol demonstrated a scalable, energy-efficient approach that enhanced safety through cyanide reduction, improved nutritional quality, accelerated drying, and increased consumer appeal. While the solar drying trials were conducted under relatively stable conditions that allowed controlled evaluation, real-world rural environments involve fluctuating irradiance, humidity, and dust contamination, which may influence drying kinetics and flour quality. Although optimized pretreatments reduced drying time and energy demand, mitigating some environmental risks, these factors remain important limitations. Further validation across diverse climates, dryer designs, and harvest seasons, as well as assessments of economic viability, life-cycle sustainability, and storage stability, are needed to confirm the robustness of this pretreatment–solar drying strategy for both rural and industrial applications.

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6. General Conclusion

This thesis presents a comprehensive investigation into the processing of cassava tubers into high-quality flour by systematically evaluating pretreatment methods and drying systems. The results demonstrate that both thermal and chemical pretreatments play critical roles in preserving the physicochemical properties of cassava flour. Blanching effectively inactivates polyphenol oxidase, thereby minimizing enzymatic browning and maintaining the flour's natural color and brightness. Chemical agents, specifically citric acid and sodium metabisulfite, inhibit oxidation reactions and expedite moisture removal from the tuber slices. Collectively, these pretreatment strategies significantly enhance sensory attributes and preserve the microstructural integrity of the resulting flour.

The assessment of drying systems revealed that oven drying, which offers precise control over temperature and duration, produces flour with optimal moisture content and high brightness. In contrast, solar drying exhibited greater variability due to its dependence on environmental conditions. Nevertheless, when combined with appropriate pretreatment, solar drying can yield flour of acceptable quality while utilizing renewable energy. Thus, the synergistic application of chemical–thermal pretreatment and controlled drying techniques is essential for obtaining cassava flour with low moisture content, high brightness, desirable texture, and preserved nutritional value.

Subsequently, optimization of pretreatment and oven-drying parameters was conducted using RSM with a CCD. The optimal drying conditions were identified at approximately 70 °C for about 10 hours, effectively minimizing moisture content while maximizing flour brightness. The ideal pretreatment protocol consisted of immersing tuber slices in a solution containing 1.31 % citric acid and 1.03 % sodium metabisulfite, followed by blanching for 1.01 minutes. This combination proved efficient in moisture extraction and also protected bioactive compounds from thermal degradation. The predictive model derived from these optimizations can be reliably applied at an industrial scale, enhancing both reproducibility and overall process efficiency.

This research also identified and validated suitable mathematical models to describe the drying kinetics of thin cassava slices. Evaluation of various thin-layer drying models indicated that the Logarithmic model best fits the oven-drying data, whereas the Wang and Singh model is most appropriate for solar drying. Validation metrics, high R^2 and low values of SSE, χ^2, and $RMSE$, confirm that these models accurately capture the water diffusion dynamics within the cassava matrix. Such predictive capability is crucial not only for process optimization but also for designing industrial drying systems, where understanding moisture loss kinetics directly impacts energy efficiency and end-product quality.

An extended evaluation involved applying the optimized pretreatment parameters to solar drying. Despite inherent variability associated with solar irradiation, the optimized pretreatment enhanced drying kinetics significantly. It increased water diffusivity and reduced activation energy, resulting in faster and more uniform drying. Flour produced under these optimized conditions also exhibited improved sensory characteristics, such as finer texture and higher consumer acceptance, while maintaining low cyanide content and better retention of essential minerals. These findings underscore that a strategically optimized pretreatment can enhance cassava flour quality even under less-controlled drying environments.

Overall, this study provides a comprehensive understanding of cassava flour processing by integrating optimal pretreatments, RSM-optimized oven-drying parameters, and validated drying-kinetics models. The approach not only improves flour quality but also bridges laboratory-scale optimization with field-relevant solar drying applications, underscoring its novelty. Sodium metabisulfite was highly effective in preserving whiteness and reducing enzymatic browning; however, its broader use may be constrained by safety concerns, regulatory restrictions, and cost, making citric acid a more practical, environmentally acceptable option for smallholder adoption. Several limitations should be noted: solar trials were conducted under relatively stable irradiance and ambient conditions, limiting generalizability across rural environments; micronutrient retention was not measured; and drying kinetics were simplified, excluding porosity effects, tissue anisotropy, and activation energy estimation under non-isothermal conditions. Moreover, the thin-layer models applied, though showing good statistical fits, did not capture dual-phase mechanisms in the falling-rate period. Future work should therefore validate this framework across diverse climates, dryer designs, and harvest seasons, while incorporating porosity-corrected and anisotropic diffusivity models, refining non-isothermal Arrhenius analysis, and applying dual-compartment models for improved mechanistic accuracy. Economic feasibility, life-cycle sustainability, and storage stability also require systematic assessment. Ultimately, these findings offer practical guidance for processors, supporting food security, reduced postharvest losses, and smallholder-oriented cassava production.

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Appendices

Appendix 1. Complete list of publications

Nainggolan EA, Banout J, Urbanova K (2024) Recent trends in the pre-drying, drying, and post-drying processes for cassava tuber: a review. Foods 13(11): 1778. https://doi.org/10.3390/foods13111778.

Nainggolan EA, Banout J, Urbanova K (2023) Application of central composite design and superimposition approach for optimization of drying parameters of pretreated cassava flour. Foods 12(11): 2101. https://doi.org/10.3390/foods12112101.

Nainggolan EA, Banout J, Urbanova K (2023) Chemical and thermal treatment for drying cassava tubers: optimization, microstructure, and dehydration kinetics. Life 13(12): 2355. https://doi.org/10.3390/life13122355.

Duque-Dussán E, Bappah M, Sanz-Uribe JR, **Nainggolan EA** (2025) Thermo-chemical characterization of coffee husk from a new variety (Coffea arabica L. var. Cenicafé 1) for biofuel production. Scientia Agriculturae Bohemica 56: 1–1. https://doi.org/10.7160/sab.2025.560101.

Anwar D, Simanjuntak EE, Sitepu I, Kinda MM, **Nainggolan EA**, Wibowo YG (2024) Thermophilic digestion of palm oil mill effluent: enhancing biogas production and mitigating greenhouse gas emissions. Jurnal Presipitasi: Media Komunikasi dan Pengembangan Teknik Lingkungan 21(3): 734–746. https://doi.org/10.14710/presipitasi.v21i3.734-746.

Yudianto D, Hanafi, Mellisani B, Ulilalbab A, Sibarani RW, Sayekti A, **Nainggolan EA** (2024) Thermal kinetic engineering of virgin red palm oil beverage product quality for storage optimization model design. International Journal of Agriculture

and Biosciences 13(4): 649–655. https://doi.org/10.47278/journal.ijab/2024.168.

Yudianto D, Ramandini VP, **Nainggolan EA**, Mellisani B, Sibarani RW, Sayekti A, Ulilalbab A (2024) Potential and simulation of functional compounds recovery from Clitoria ternatea L. extract during the commercial sterility process. International Journal of Experimental Research and Review 42: 262–277. https://doi.org/10.52756/ijerr.2024.v42.023.

Appendix 2: **Conferences attended**

Nainggolan EA, Urbanová K (2024) A review of dehydration kinetics of cassava tubers in solar drying. The 5th International Multidisciplinary Conference for Young Researchers, 03 – 04 October 2024, Prague, Czech Republic.

Nainggolan EA, Anwar D, Sariati MN, Kinda MM, Urbanová K (2023) Influence of chitosan and glycerol-sorbitol concentrations on physical properties of palm oil empty fruit bunches-based bioplastics. The 4th International Multidisciplinary Conference for Young Researchers, 05 – 06 October 2023, Prague, Czech Republic.

Nainggolan EA, Anwar D, Urbanová K (2024) A model-based study of the dehydration kinetics of pretreated cassava flour. IOP Conference Series: Earth and Environmental Science 1352(1): 012096. https://doi.org/10.1088/1755-1315/1352/1/012096. The 5th International Conference on Natural Resources and Technology, 29 – 30 August 2023, Medan, Indonesia.

Nainggolan EA, Anwar D, Urbanová K (2022) Effects of soaking and blanching on drying kinetics of cassava flour. The

4th International Conference on Food Science and Engineering, 11 – 12 October 2022, Surakarta, Indonesia.

Nainggolan EA, Urbanová K (2022) Mathematical modelling of oven drying kinetics of pretreated cassava flour. Tropentag, 14 – 16 September 2022, Prague, Czech Republic.