AKENTEN APPIAH-MENKA UNIVERSITY OF SKILLS TRAINING AND ENTREPRENEURIAL DEVELOPMENT

EXTRACTION AND CHARACTERIZATION OF MICROCRYSTALLINE CELLULOSE FROM MALTED SORGHUM MASH FOR PHARMACEUTICAL USE

N-TEYABI BOSCO NYOBON

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EXTRACTION AND CHARACTERIZATION OF MICROCRYSTALLINE CELLULOSE FROM MALTED SORGHUM MASH FOR INDUSTRIAL USE

BY

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DECLARATION

Candidate's Declaration

I hereby declare that this thesis, with the exception of quotation and references contained in published works which have been duly acknowledged; is the result of my own original work and that no part of it has been presented for another degree in this university or elsewhere.

Candidate's Name: N-TEYABI BOSCO NYOBON

Signature: …… Date:……

Supervisors' Declaration

We hereby declare that the preparation and presentation of the thesis were supervised in accordance with the guidelines on Supervision of thesis laid down by the Appiah-Menka University of Skills Training and Entrepreneurial Development.

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DEDICATION

I dedicate this work to the Almighty God, whose grace, wisdom and strength guided me through every step of this work. To my family, for their unwavering support, prayers and encouragements and to every student who dares to dream, work hard and rise above challenges.

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LIST ABBREVIATIONS

BP	-	British Pharmacopoeia
CI	-	Compressibility Index
CrI	-	Crystallinity Index
DSC	-	Differential Scanning Calorimetry
FTIR	-	Fourier-transform infrared spectroscopy
HR	-	Hausner Ratio
MCC		Microcrystalline cellulose
MSM	-	Malted Sorghum Mash
SD	-	Sorghum Derived
SEM	-	Scanning Electron Microscopy
TGA	-	Thermogravimetric Analysis
USP	-	United States Pharmacopoeia

ABSTRACT

Microcrystalline cellulose (MCC) is a multipurpose polymer used in biomedical, food, chemical, and cosmetic industries. Though malted sorghum is widely utilized in different agro-industries, its mash is often treated as an agricultural waste. This study aimed to extract MCC from malted sorghum mash (MSM), characterized it and study its use as a sustainable source of cellulose for MCC production. The extraction process involved sequential treatment of the malted sorghum mash with water, 4% (w/v) NaOH solution, 5.5% (v/v) sodium hypochlorite for bleaching, 40% (v/v) H_2O_2 solution for further bleaching and 3.0M hydrochloric acid for acid hydrolysis. From the study, MCC was successfully extracted from malted sorghum mash. Fourier transform infrared analysis of the extract confirmed the absence of impurities in the MSM-MCC fibers produced. The resultant MSM-MCC fiber displayed a distinct crystalline cellulose structure, as indicated by X-ray diffraction analysis of MSM-MCC product reported crystallinity index of 86.63%. Scanning electron microscopy (SEM) analysis showed variations in the morphology of the MSM-MCC product whilst Thermogravimetric analysis (TGA) of the MSM-MCC product also demonstrated enhanced thermal stability for the product. MSM-MCC extracted had a percentage yield of 60%. The MSM-MCC was insoluble in distilled water, dilute aqueous hydrochloric acid, acetone and ethanol. This insolubility is consistent with the typical behaviour of cellulose-based material. MCC production from malted sorghum mash presents promising opportunities for use as an emulsifier, stabilizer, and thickener in the chemical, pharmaceutical, and food production sectors.

CHAPTER ONE

INTRODUCTION

This chapter introduces the concept of microcrystalline cellulose (MCC), highlighting the relevance of using malted sorghum mash as a potential alternative raw material. It outlines the background, statement of the problem, objectives of the study, research questions, research hypothesis, significance of the study, and the scope and limitations.

1.1 Background to the Study

Microcrystalline cellulose (MCC) is a white, and fine-particle powder derived from cellulose (Zhang et al., 2019). It is widely used in pharmaceuticals, food, and cosmetic industries, due to its excellent binding, thickening, and stabilizing properties (Randis et al., 2023).

The MCC is characterized by its fine, crystalline particles formed from acid hydrolysis of cellulose fibres (Zhang et al., 2019). The extraction process involves treating cellulose with 2.5M hydrochloric acid. This results in the partial depolymerization of cellulose fibres while retaining their crystalline structure (Agboeze et al., 2022). This crystalline structure contributes to the desirable properties, such as stability, mechanical strength, and compatibility with various formulations (Tarchoun et al., 2019).

The global MCC market has been expanding annually and this is due to the rising preference for natural and biodegradable materials in the food and pharmaceutical sectors (Agboeze et al., 2022).

The conventional sources of MCC are materials of plant origin which include wood pulp and cotton (Abdul Khalil et al., 2018). However, these sources are unsustainable due to deforestation and high land-use requirements (Gichuki et al., 2022). The increasing demand for sustainable, cost-effective and eco-friendly materials necessitates an exploration for alternative sources for MCC production (Abu-Thabit et al., 2020). According to Hachaichi et al., (2021), malted sorghum mash, which is a by-product of the brewing industry, is a promising substrate for MCC production. Due to its high cellulose content, it has not been explored and is hence underutilized.

Sorghum, a drought-resistant cereal grain, has not been recognized as a potential alternative source of MCC globally (Tan et al., 2023). This cereal is cultivated in many regions (Gabriel et al., 2020). It thrives in arid areas where other crops may die (Mohamad et al., 2013). The malted sorghum mash, a by-product of the brewing process, contains a considerable amount of cellulose together with other beneficial components such as hydrolytic enzymes, proteins, antioxidants, vitamins and minerals among others (Hermin et al., 2017). These make it an excellent candidate for MCC extraction (Azum et al., 2021).

Sorghum is a staple food in many developing countries (Wahib et al., 2021). Increasingly, it has been recognized for its nutritional value and functional properties (Husin et al., 2016). Sorghum contains high levels of dietary fibre, antioxidants, and essential nutrients (Abu-Thabit et al., 2020). These make it a valuable ingredient in food formulations (Akinoso et al., 2022).

Additionally, this by-product of sorghum represents a significant portion of waste in the brewing industry, and it necessitates innovative protocols for its utilization (Dyah et al., 2023).

A recent study has explored MCC extraction and found it to be ubiquitous in non-wood sources (Zeleke et al., 2022). The study highlighted the potential of agricultural residues and by-products as sustainable sources for MCC extraction (Athirah et al., 2020). For example, malted sorghum mash is rich in cellulose and presents an attractive alternative for MCC production (Beroual et al., 2021). The malted process, which involves germination of the grains, not only enhances the nutritional profile of sorghum; but it also alters its structural properties, thereby improving the yield and quality of MCC extracted (Katakowala et al., 2020).

Malted sorghum mash is obtained from the malting process, which involves; soaking the grains in water to initiate germination, followed by drying the soaked grains. This process enhances the nutritional profile of sorghum, increases its enzymatic activity and increases its carbohydrate availability (Misgana et al., 2019). After fermentation, the residual mash is rich in cellulose, and the cellulose can be extracted to produce MCC (Athirah et al., 2020). Leveraging malted sorghum not only addresses waste management issues in the brewing industry; but also promotes utilization of a sustainable biomass source to produce viable raw materials for other industries (Senusi et al., 2020).

Globally, considerable quantities of malted sorghum mash are produced daily by sorghum beer production (Liu et al., 2018). These organic wastes can contaminate the environment; if not managed properly (Abdul Khalil et al., 2018). Therefore, effective management of this biodegradable waste to reduce its negative impacts on the environment is needed. These wastes when processed, could help achieved high economic benefit (Fathi et al., 2018).

In Ghana, the ever-growing sorghum beer industries correspond to increased demands for labour. Despite its untapped use as a feed source for pigs, malted sorghum mash has not been studied scientifically to determine ways it could be used in other economically viable ventures (Belali et al., 2019). Environmental monitoring agencies and sorghum beer brewers need to collaborate to determine efficient and ecofriendly protocols to handle this industrial by-product (Kian et al., 2020).

Their collaborations must include recycling malted sorghum mash and other waste materials of plant origin for higher economic gains and effectively combat contamination issues often aimed at untapped agro-wastes. Economically, these agro-waste could be an alternative source of raw materials for MCC production. Environmental issues that originate from plant-based materials due to their organic characteristics could massively pollute the environment when released into it (Belali et al., 2019).

Investigation on MCC extraction, from industrial waste such as coconut husk, pineapple leaves, sugarcane bagasse, banana rachis, cornstalks, wheat straw, cotton waste; among

others, has been done (Athirah et al., 2020). The use of industrial by-products of plant-based materials as raw materials for MCC extraction not only helps provide affordable and renewable resources of materials but also helps to deal with waste management problems (Liu et al., 2018).

How waste is produced from industries and other biodegradable activities is managed is vital with respect to greenhouse emissions. Disposal of industrial waste from local industries, such as the sorghum beer industry; impacts several health issues and contributes to disease outbreaks. Thus, there is a need for alternative eco-friendly waste management protocols which would be beneficial to individuals and the nation (Trachi et al., 2016).

1.1.1 Extraction of Microcrystalline Cellulose from Alpha Cellulose

Huge amounts of biodegradable waste are produced in industrial processes of agro-products (Athirah et al., 2020). Most of this waste is either used to feed animals or released into the environment as a way to control such industrial waste (Hermin et al., 2017). However, higher quantities of this waste are biodegradable and have high cellulose content. However, this waste should not be seen as waste as it could be a source of raw materials for other industrial applications (Haque et al., 2021).

Cellulose is among the most abundant natural biopolymers known (Husin et al., 2016). Cellulose is an important source for renewable polymers used in producing cellulose derivatives such as micro: and nano-crystalline cellulose, among others (Kalita et al., 2013). The occurrence of crystalline structures and amorphous regions in cellulose is

hydrolyzable under acidic conditions to eliminate these structures in cellulose (George et al., 2015). This acid hydrolysis treatment of cellulose produces molecules of lower molecular weight, nano and micro size particles (Husin et al., 2025).

Cellulose is also considered to be a linear polymer of carbohydrate (Nazir et al., 2023). It is made up of β-D glucopyranose monomers, and each monomer has three hydroxyl groups (Kharismi et al., 2018). Thus, cellulose molecule with increased functionality is given (Agboeze et al., 2022). The nature of such cellulose includes chirality, hydrophilicity, and biodegradability of cellulose, which have been thoroughly studied (Kalita et al., 2013).

Cellulose and its derivatives have been thoroughly been investigated due to their mechanical, chemical and biological properties (Kian et al, 2017). Cellulose and its derivatives have been applied in several processes including pharmaceuticals, paper production, cosmetics and biomaterials production among others (Klemm et al., 2011). These compounds are very stable due to the occurrence of wide networks of hydrogen bonds (Karna et al., 2021) and have relatively high melting points (Qinfeng et al., 2018). Cellulose materials possess good elasticity and flexibility properties (Ohwoavworhua et al., 2010). These properties permit them to have elevated quantities in processes which require their use (Okoye et al., 2015). For instance, α-cellulose, when purified, is insoluble in 17.50% sodium hydroxide solution, while the impure hemicellulose and β-cellulose are very soluble in processes involving acid hydrolysis (Olugbenga, 2014). Materials with cellulosic characteristics need to be treated to alter their structures prior to their usage (Naduparambath et al., 2016). This improvement in their structures permit them not to take

part, easily in reactions (Rasheed et al., 2020). Thus, these improved structures make them important compounds when they are being converted into chemicals and other fuels (Fouad et al., 2020).

Prior to their involvement in the production processes, cellulose and its derivatives are treated via physical, chemical or a combination process. The Chemical treatment takes place in an acidic or alkaline medium to aid the breakdown of their structures and yield increased amounts of product (Rojas et al., 2012). Sulphuric acid and sodium hydroxide are the most commonly used acids and bases in the chemical treatment process of cellulose and its derivatives. Other researches have used nitric, hydrochloric and phosphoric acids (Sainorudin, 2018).

MCC extraction comprises numerous vital stages, including prior-treatment, hydrolysis, and purification steps. Prior-treatment breaks down the composite matrix of the raw material via chemical or enzymatic protocols (Hermin et al., 2017). The hydrolysis stage also employed acid or enzymatic treatments to depolymerize cellulose into micro particles (Singh et al., 2017).

The extraction of MCC from lignocellulosic biomass by acid hydrolysis improves its applicability in processes which require its applications (Mohamed et al., 2013). Acid hydrolysis effectively breaks down cellulose into microcrystals while preserving desirable structural characteristics of the material (Tan et al., 2023). The process of acid hydrolysis involves the treatment of cellulose extract with either dilute or concentrated acids (Randis

et al., 2023). This enables the cleavage of the glycosidic bonds through protonation. This selective degradation of cellulose extract allows for the removal of amorphous parts of cellulose, which are more susceptible to acid action. This helps to produce crystalline end-products as indicated by Liu et al (2020).

The efficiency of the selective degradation process is influenced by several factors, including but not limited to the strength of the acid, reaction temperature, duration of the reaction, and the type of cellulose used. Optimization of these parameters, namely, acid strength, temperature of the reaction medium, and duration of the reaction process as reported by Agboeze et al., (2022) is vital to maximized MCC yield and minimized the formation of by-products.

The acid hydrolysis procedure is straightforward and can be scaled for industrial applications (Agboeze et al., 2022). The use of acids such as hydrochloric or sulphuric acid not only enhances the solubility of cellulose but also facilitates recovery of the by-products, such as glucose and other oligosaccharides (Tessema et al., 2023).

The resultant MCC exhibits improved flow properties such as; reduced angle of repose, lower-cohesiveness, compressibility index, decreased moisture content, better powder uniformity and stability. These properties make the MCC produced suitable for use in drugs formulations, especially tablets and as food additives (Alebiowu et al., 2003). Recently, advances have been made in acid hydrolysis techniques including microwave-assisted and

ultrasound-assisted protocols. These advancements in the acid hydrolysis of cellulosic materials, according to Azum et al., (2021), have improved efficiency of MCC extraction.

These innovations have decreased processing times and energy consumption which align with the growing demand for eco-friendly manufacturing practices (Fouad et al., 2020). As the field of acid hydrolysis-based MCC production evolves, research is done on optimization of the conditions of acid hydrolysis, and different sources of biomass could be applied in an attempt to obtain sustainable MCC production (Asif et al., 2022).

1.1.2 Importance of Microcrystalline Cellulose

MCC is a cellulose derivative that has recently gained significant interest in the research world due to its huge potential industrial applications and distinct physicochemical properties (Romruen et al., 2020). To produce high-quality MCC, the amorphous regions of the cellulose must be eliminated either by chemical or physical process or both to obtain micro particles with a well-arranged crystalline structure (Asif et al., 2022). This well-crystalline structure has significant thermodynamic and mechanical properties, which make it usable for use as an additive in food, cosmetics and pharmaceutical industries (Zeleke et al., 2022).

In the pharmaceutical industry, MCC is employed as an excipient in drug formulation (Romruen et al., 2020). It facilitates drug manufacturing processes and helps improve the physical and chemical properties of drugs (Romruen et al., 2020). As an excipient, MCC can be used as a binder, disintegrant, filler, and flow aid in pharmaceutical processes (Liu

et al., 2025). This ensures that drugs produced have uniform composition, and stability, which helps to regulate drug formulation (Uzondu et al., 2022).

In the food industry, MCC is valued for its functional properties like emulsification, stabilization and dietary fibre contents that support digestive health (Zeni et al., 2016). In the cosmetic industry, MCC has gained attention (Romruen et al., 2020). MCC acts as a thickening agent to help improve product texture and skin compatibility (Liu et al., 2018). The multifunctional nature of MCC makes it an important ingredient for increasing industrial demands for safe, effective and sustainable products (Zhao et al., 2018). Recently, research has found new uses for MCC (Romruen et al., 2020). It has been found to improve product quality and consumer safety (Zeleke et al., 2022).

MCC is employed as an anti-caking agent, stabiliser, and texture enhancer in the food and beverage industries (Fouad et al., 2020). MCC can absorb water. This makes it an important substance in the production of powdery products such as spices, seasoning mixes, and powdered drinks (Trache et al., 2017). This helps prevent clumping of final products. MCC also helps to improve the texture and taste of food products by enhancing their water-binding properties (Tessema et al., 2023).

According to Kannusa et al (2018), MCC has been widely used as an important compound in the cosmetic industry due to its properties, such as mattifying effect, eco-friendliness, high absorbency, among others and the benefits of a safe and mild polishing effect. In the cosmetic industry, MCC is employed as a thickening agent due to its ability to absorb water

and produce uniformity in the thickened products (Trache et al., 2016). In the cosmetic industry, MCC is used in creams, lotions, and gels to help produce fine texture and stable products as indicated by Uesu et al., (2000). MCC is also used as a binding agent in the pharmaceutical industry to help hold products firmly to prevent separation of the components of the products (Azum et al., 2021).

Furthermore, MCC is also used as a bulking agent to help increase the volume and bulkiness of cosmetic products (Ul-Islam et al., 2012). This increased bulkiness in products gives the products improved spreadability (Fouad et al., 2020). MCC also has the capacity to absorb excess oil on the skin used in cosmetic production. Its absorbing property makes it applicable in mattifying creams or oil-controlled powders formulation (Veeramachineni et al., 2016).

According to Kunusa et al (2018), no considerable variation exists between MCC produced using different wood pulps as raw materials. Hard-woods polymerized slowly compared to soft-woods (Kunusa et al., 2018). Thus, MCC powder obtained from hard-woods has a lower crystalline structure than that obtained from soft woods (Kunusa et al., 2018). These structural differences show that hydrolysis occurs at both the crystal and amorphous regions of the wood pulp during MCC extraction (Kunusa et al., 2018). However, small differences exist in the kinetic parameters of two MCC products extracted from soft and hard wood pulps (Kunusa et al., 2018).

MCC has a large breaking strength and rigidity which makes it a suitable reinforcement filler in bioplastics and bio-based materials. It improves the mechanical properties of polymer materials and promotes lightweight, renewable materials (George et al, 2015).
The MCC's high surface area and insolubility in water; contribute to the steadying of emulsions and suspensions in dairy products, sauces, beverages, and desserts (Siro et al., 2010).

The exploration of MCC in hydrogels, tablet delivery systems, and scaffolds for tissue engineering is due to its biological compatibility, high surface morphology, and ability to form porous structures. Research has demonstrated that MCC can be mixed with polymers to help cell bonding and growth. This makes it suitable for cell-based therapy (Ul-Islam et al., 2012).

MCC has its most vital uses in the pharmaceutical industry as a direct binder and diluent in drug compression. MCC offers perfect flowability and compressibility, making it suitable for tablet synthesis without reliance on wet granulation (Akinoso et al., 2022). MCC's high binding ability enhances the mechanical strength and uniformity of pills. MCC ensures the breakdown due to its capillary action and wicking ability. It absorbs water quickly, ensuring drug disintegration and release (Lieberman et al., 1990). Additionally, the chemical stability and inert nature of MCC make it compatible with a large range of pharmaceutically active ingredients (Akinoso et al., 2022).

MCC also serves as a non-caloric bulking agent, which can replace fat or sugar in low-calorie foods without texture alteration. Moreover, it enlarges dietary fibre amounts, enhancing gastrointestinal wellness and appetite satisfaction (Saeed et al., 2020). MCC is employed in toothpastes, creams, and lotions as a gelling agent, polishing agent, and emulsifier. It enhances the texture, improves flow properties, and gives a smooth feel on application (Karna et al., 2022). Its toxic-free and irritating-free nature qualifies it as an ideal candidate for skin-sensitive production. MCC is produced from renewable organic materials and is entirely biodegradable, making it an eco-friendly option to synthetic polymers in-industrial uses, such as packaging, paper strengthener, and green multi-phase-materials (Trache et al., 2017). The hygroscopic character of MCC significantly impacts its flow behaviour. The retained moisture in MCC increases cohesion and decreases flow. Hence, preserving the ideal storage conditions is key for maintaining its functional properties (Zeleke et al., 2022).

Globally, MCC extraction from wood is continuing, and the practice has negatively affected; the global effort to obtain alternative sources of renewable energies from plants (Kunusa et al., 2018). Malted sorghum, a cereal grain rich in cellulose, provides a sustainable and abundant substrate for MCC production. This work investigates physico-chemical parameters of MCC extracted from malted sorghum mash. The work also seeks to maximize the yield and the purity of MCC extracted from malted sorghum mash (Agboeze et al., 2022).

This study will also investigate structural characteristics, including its crystallinity, particle size, and surface area morphology. Those are particularly important because they influence the applicability of MCC in various pharmaceutical formulations (Azum et al., 2021). This study contributes to the establishment of MCC derived from malted sorghum mash. It further contributes to knowledge on sustainable materials for the pharmaceutical industry. Thus, this work paves the way for more environmentally friendly production protocols of MCC for industrial use (Agboeze et al., 2022).

1.1.3 Characterization of Extracted Microcrystalline Cellulose

Characterization of the extracted MCC is essential to understand its physical and chemical properties as those properties dictate its usage in various applications (Azum et al., 2021). Important characteristics of MCC include particle size, surface area and morphology, crystallinity index, and thermal stability as documented by Agboeze et al., (2022).

Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), thermogravimetric Analysis (TGA and differential Scanning Calorimetry (DSC) are commonly employed techniques used in analyzing properties of MCC (Randis et al., 2023).

Structural properties of MCC derived from malted sorghum mash are important as they attribute significant effects on its performance in food and also pharmaceutical applications (Azum et al., 2021). For instance, the extent of crystallinity in MCC affects its solubility and bioavailability and thus makes it vital to tailor extraction processes which optimize the

properties of MCC (Agboeze et al., 2022). In various applications, the character of MCC ensures its quality and consistency. The quality attributes of MCC include crystallinity index, particle size, structural morphology, and moisture content (Agboeze et al., 2022). Each of these attributes affects the behaviour of MCC and its applications in pharmaceutical formulations, such as tablet compression, stability, and drug release profiles (Zeleke et al, 2022).

Characterization of an extracted MCC using FTIR, XRD, SEM, and particle size analysis is vital for optimizing its usage in various applications (Azum et al., 2021). Understanding the physical and chemical properties of MCC allows for its improved formulation strategies and ensures that its use as an excipient in drug formulation meets the required quality standards. Thus, continued research on MCC extraction, characterization and its applications will contribute to the development of more efficient and effective pharmaceutical products as indicated in a report by Randis et al., (2023).

MCC's characterization involves a set of analytical protocols to explain its chemical structure, crystalline nature, morphology, and solubility behaviour: FTIR analysis is used to verify the removal of amorphous components of cellulose, such as lignin and hemicellulose and to confirm the retention of native cellulose functional groups. For example, the absence of peaks at 1733 cm^{-1} aligned to lignin/hemicellulose C=O stretching indicates a highly purified MCC (Udo et al., 2020). The occurrence of peaks at hydroxyl (OH), C–H, and glycosidic (C–O–C) vibrations further confirms the cellulose structure as reported in a study by Temitope et al., 2023).

XRD is utilized to evaluate the crystallinity of the extracted MCC. Specific diffraction peaks close to 15° and 22° (2θ) conform to cellulose I polymorphs, and the crystallinity index (CrI) is determined using the Segal protocol, revealing a clear understanding of the degree of crystallinity of the MCC structure (Asif et al., 2022;). SEM demonstrates the morphology and particle properties of MCC. MCC usually occurs as microfibrils or granular crystals, based on the material origin. For instance, MCC produced from roselle fibres showed rough surface morphology and little aggregation (Udo et al., 2020).

Protocols such as Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) also evaluate thermal stability and moisture content of the extracted MCC. TGA mostly shows initial weight loss near 100 °C corresponding to moisture and main decomposition at a temperature range of 250–330 °C for the synthesized MCC (Udo et al., 2020).

The flow properties of MCC are largely based on its moisture content, shape, particle size, and surface texture. The poor flowability of MCC is generally due to the irregular and porous nature of the particles, while more granular MCC powders contribute to excellent flowability. Smaller particle sizes have greater interparticle forces, which aid them exhibiting more cohesiveness (Okoye et al., 2015).

Research has assessed MCC using standard flow indicators like Carr's Compressibility Index (CI), crystallinity index (CrI) and Hausner Ratio (HR). In a report by Kian et al., (2017), a Carr's index ranging from 5–15% demonstrates excellent flow, whilst values greater than 25% indicate poor flow. MCC generally shows CI and HR values suggesting

moderate to passable flow, depending on the raw material source and processing protocol employed. For example, native MCC usually have CI values falling between 15–23% and HR values in the range of 1.2–1.4 as indicated in a study by Uzondu et al., (2022).

MCC's flowability is closely attributed to its tapped and bulk densities. Low bulk density of MCC between 0.2–0.4 g/cm^3 and high packing ability usually limit its flow. These characteristics can be enhanced via particle engineering protocols like co-processing or granule formation. Co-processed MCC demonstrates improved flow because of the particle size increase and decreased cohesiveness (Rojas et al., 2012).

Angle of repose of MCC is an important index. MCC normally demonstrates an angle of repose in the range of 30° - 40°, showing fair flow properties (Alebiowu et al., 2003). However, enhancements like spray drying or combining with glidants such as colloidal silicon dioxide have been demonstrated to decrease the angle of repose and improve flowability. The hygroscopic character of MCC significantly impacts its flow behaviour. The retained moisture in MCC increases cohesion and decreases flow. Hence, preserving the ideal storage conditions is key for maintaining its functional properties (Rojas et al., 2012).

1.2 Statement of the Problem

Extraction of MCC from wood pulp and use as an excipient in drug formulations is an important issue in formulation science (Randis et al., 2023). Globally, industrial demands for MCC have soared rapidly due to the emergence of new diseases (Agboeze et al., 2022).

Though MCC is mostly used as an excipient, it can also be a source of raw material in the production of cellulose nano-particles (Azum et al., 2021). However, MCC extraction from malted sorghum mash has not been hugely investigated as reported by Zeleke et al, (2022). Thus, there exists limited knowledge on its characterization for use in pharmaceutical formulations and as an absorbent in cosmetic industry, among other applications (Fouad et al., 2020). Limited works on the physical identification of MCC extracted from malted sorghum mash have blocked its potential as a cheap and renewable source of raw material for industries that require MCC in their protocols (Azum et al., 2021). Hence, addressing this research gap will not only broaden the scientific understanding of alternative MCC sources but also support the development of sustainable and locally available materials to meet the growing industrial demand.

1.3 Significance of the Study

This research is significant for the following reasons:

1 It will add to the growing body of knowledge regarding sustainable alternative sources of cellulose for industrial applications (Agboeze et al., 2022). The study will address environmental issues associated with traditional sources of MCC and will promote economic development in the brewing industry (Athirah et al., 2020a).

2 The successful extraction and characterization of MCC from malted sorghum mash can lead to new applications in various sectors and reduce reliance on synthetic MCC.

1.4 Objectives of the Study

The main objective of this study is to investigate the by-product of sorghum beer and its potential to be employed as MCC via an acid hydrolysis protocol. This current study also looks at the likelihood of using the by-product of sorghum beer production as a sustainable and cheap precursor for MCC products. This study is important because wood pulp used in MCC extraction could cause deforestation and severe environmental pollution. Specifically, this current work will extract MCC from malted sorghum mash and investigate its properties for satisfactory industrial use.

1.4.1 Specific Objectives of the Study

To achieve this main objective, the specific objectives of this work are to:

1 extract MCC from malted sorghum mash by the acid hydrolysis method.

2 study the surface morphology of the extracted MCC by Fourier-transform infrared spectroscopy (FTIR).

3 examine surface morphology, particle size, and microstructure of the MCC extracted by Scanning Electron Microscopy (SEM).

4 Determine the crystal structure, crystallinity, and phase composition of the MCC extracted using the X-ray diffraction (XRD) technique.

5 examine the thermal stability and decomposition profile of the extracted MCC using DSC-TGA analysis.

1.5 Research Questions

1 What are the best conditions for extracting MCC from malted sorghum mash?

2 How does pre-treatment of malted sorghum mash affect MCC yield and quality?

3 How does the MCC yield from malted sorghum mash compare to other sources?

4 What are the key physical and chemical properties of extracted MCC?

5 What is the crystallinity index and particle size of the extracted MCC?

6 How does sorghum-based MCC differ from commercial MCC structure?

7 Does the extracted MCC meet the industrial quality standards?

8 Is MCC production from malted sorghum mash economically viable?

9 What environmental benefits does this source offer?

10 Can the extracted MCC be modified for advanced applications?

11 What industries can best use MCC from malted sorghum mash?

1.6 Research Hypothesis

1 HO: MCC extraction from malted sorghum mash is possible and would produce considerable quantities of modified cellulose micro particles.

1.7 Justification of the Study

Extraction and characterization of MCC from malted sorghum mash represents a promising avenue for the production of sustainable biopolymer (Azum et al., 2021). As the global demand for natural and environmentally friendly materials continues to rise, this research not only aims to explore the potential of malted sorghum mash as a cellulose source but also contributes to broader discourse on resource utilization and waste

management (Athirah et al., 2020). By focusing on the extraction and characterization processes of MCC extraction from malted sorghum mash, this study seeks to unlock the potential of malted sorghum mash and pave the way for its application in diverse fields.

MCC is employed as an excipient in pharmaceutical drug formulations particularly due to its compressibility, binding properties, and flowability among others. It is also used in the cosmetic industry due to its ability to absorb excess oil from the skin. MCC extraction from malted sorghum mash would reduce the operational cost of pharmaceutical and cosmetic compounds due to its sustainable nature and as a cheap source of raw material for use in those industries.

Malted sorghum mash application as a source of MCC promotes environmental sustainability since it would reduce waste generated by these industries, particularly the brewing industry (Fouad et al., 2020). Knowledge of properties of the MCC extracted from malted sorghum mash would ensure quality and consistency of pharmaceutical drugs, particularly tablets. Characterization of MCC would offer great insight into its chemical composition, particle size distribution, and surface morphology of MCC.

CHAPTER TWO

LITERATURE REVIEW

This chapter examines existing research on MCC, focusing on its sources, properties, extraction protocols and its industrial applications. The chapter further explores the industrial applications of sorghum and its by-products. It highlights various characterization techniques such as FTIR, SEM, XRD, DSC, solubility and flow analysis. Finally, the chapter outlines research gaps and sets the practical and theoretical framework for the current study.

2.1 Microcrystalline Cellulose

MCC is an excipient employed in pharmaceutical drug formulations and as an excess oil absorbent in cosmetics and food industries due to its wide spread availability, biocompatibility, inertness, and excellent compressibility properties (Azum et al., 2021). It is derived from natural cellulose sources and a partially depolymerized cellulose material with small crystalline particles and high purity (Azum et al., 2021).

MCC is mostly produced by acid hydrolysis of cellulose-rich plant materials, including wood pulp and cotton linters (Agboeze et al., 2022). MCC has a uniform size distribution with its dimensions ranging from 5 to 50 microns (Zeleke et al., 2022). MCC is a pristine, white, non-reactive, odourless powder with crystalline properties (Azum et al., 2021). It is derived by partial depolymerization of α-cellulose using mineral acids and refining to eliminate impurities (Agboeze et al., 2022). MCC is recognized for its exceptional properties and continues to be the most commonly utilized excipient for direct compression

in pharmaceutical tablets (Agboeze et al., 2022). Additionally, MCC serves as a robust dry binder, tablet disintegrant, absorbent, filler or diluent, lubricant, and antiadherent in food, cosmetics and pharmaceutical industries (Getachew et al., 2023).

MCC, derived from α-cellulose through partial depolymerization, has alpha bonds that resist digestion in the human body due to the absence of specific enzymes (Getachew et al., 2023). MCC is a tasteless, odourless white crystalline powder, ideal for various industries like pharmaceuticals, food, and cosmetics, as indicated by Agboeze et al., (2022). The strength, fibrous texture, crystalline structure, lightweight nature, biocompatibility, water insolubility, and biodegradability of MCC makes it a preferred excipient in the modern era of sustainable materials with diverse application characteristics (Azum et al., 2021).

Due to increasing environmental issues, materials are being refined to be eco-friendly and biodegradable in nature. However, conventional production of MCC from wood pulp and purified cotton poses ecological challenges as reported by Agboeze et al., (2022). Over the past two decades, efforts have been directed at extracting MCC from natural sources and plant waste that are both environmentally friendly and cost-effective to produce materials with high cellulose content (Belali et al., 2019). Insolubility of MCC in both water and organic solvents as well as its inertness in majority of chemicals enable it to be employed as a potential excipient in pharmaceutical drugs formulations (Getachew et al., 2023). These properties of MCC allow it to be employed as fillers and binders in pharmaceutical tablets (Getachew et al., 2023).

MCC is very stable and it does not interact with other constituents of a pharmaceutically formulated drug such as Tablets (Agboeze et al., 2022). So, when MCC is employed in pharmaceutical productions, it would not affect the stability and efficacy of the drug formulation and production (Getachew et al., 2023).

2.2 Raw Materials for Microcrystalline Extraction

There are diverse raw materials used in MCC extraction and purification protocols (Belali et al., 2019). The extraction and purification protocols employed influence properties and functions of the resulting product (Belali et al., 2019). Every material has distinct benefits and limitations. Among these, agricultural residues of plant origin stand out as a viable and eco-friendly alternative to wood and they offer both economic advantages and environmental sustainability (Agboeze et al., 2022). According to Zeleke et al., (2022), research into more sustainable MCC extraction protocols, such as enzymatic hydrolysis and physicochemical processes, is ongoing. These studies aimed to improve the practicality of producing MCC from a wide array of raw materials.

Raw material selection for the MCC extraction protocol depends on factors such as the desired quality of the final product, cost-effectiveness, and potential impact on the environment. According to Zeleke et al., (2022), attention has been focused on minimizing waste and promoting sustainability in MCC production. Almost any material with high cellulose content, is suitable for MCC production (Belali et al., 2019). MCC production involves cellulose extraction from sources, such as wood and other agricultural waste of plant origin (Agboeze et al., 2022). Each raw material has a unique character and chemical

composition that affects the properties of the final product. Several steps are involved in the MCC extraction process. These steps, include pretreatment, delignification, and purification. These when done well improve the quality of the MCC extracted.

Wood Pulp is the most common source for MCC production. Wood pulp is primarily derived from softwood (pine) or hardwood (birch, beech) trees (Randis et al., 2023). The cellulose fibres in wood pulp are processed to remove lignin and hemicellulose to obtain purified cellulose that can be further processed into MCC (Randis et al., 2023). Wood, particularly from coniferous and deciduous trees, is a primary source for MCC extraction (Randis et al., 2023).

The cellulose content in wood varies significantly, with hardwoods generally containing about 40-50% cellulose and softwoods containing about 45-55% cellulose (Singh et al., 2017). Also, agricultural residues such as rice husks, wheat straw, and corn stover have gained attention as sustainable raw materials for microcrystalline cellulose production (Sainorudin et al., 2018). These residues are abundant and often underutilized. For instance, Saini et al. (2016) demonstrated that rice husks, which contain about 35-40% cellulose, can be converted to MCC through a two-step chemical process involving sodium hydroxide followed by hydrochloric acid treatment. Equation for the reaction is shown below;

Lignocellulosic biomass (rice-husk) + $NaOH_{(aq)} \longrightarrow$ Cellulose (purified)+soluble lignin/hemicellulose 1

$(C_6H_{10}O_5)_n$ (Purified Cellulose) $+HCl_{(aq)} \longrightarrow$ MCC + Soluble sugars 2

Various plant fibres, such as cotton and jute, also serve as excellent sources of microcrystalline cellulose (Saulnier et al., 2007). Cotton has a high cellulose content (90% or more) and provides a high yield of microcrystalline cellulose with desirable properties (Shi et al., 2018).

The extraction process typically involves bleaching and acid hydrolysis to purify the cellulose (Randis et al., 2023). The second most common raw material for MCC extraction is cotton linters. These are short fibres that adhere to cotton seeds after the longer fibres used in textile production are removed (Liu et al., 2018). These fibres are almost pure cellulose and are another common source for MCC production. These are cotton fibres valued for their high purity and uniformity (Liu et al., 2018).

Algal biomass of certain species of green algae, has emerged as a novel source for MCC production (Karna et al. 2022). Studies by Karna et al. (2022) indicate that algae can yield cellulose through enzymatic hydrolysis, and this offers a more environmentally friendly alternative compared to traditional sources (Liu et al., 2018). The cellulose extracted from algae exhibits unique properties, which can be advantageous in specific applications (Sonia et al. 2013).

Bamboo, with its rapid growth and high cellulose content of about 55% has been identified as a promising raw material for MCC extraction (Zhang et al. 2019). Research by Zhang et al. (2019) showed that bamboo-derived MCC can be produced efficiently through a combination of mechanical and chemical treatments to yield MCC with superior

crystallinity and purity. The exploration of various raw materials for MCC extraction shows that they have potential for sustainable and efficient production of MCC (Agboeze et al., 2022). Existing research has focused on optimizing extraction processes and characterizing the properties of MCC derived from alternative sources to enhance its applicability across diverse industries (Agboeze et al., 2022).

Prior research indicates that cellulose derived from various sources and hydrolytic conditions varies in the overall attributes of MCC including factors like particle size and aggregation tendencies (Zhao et al.,2018).

Sorghum (*Sorghum bicolor* L. Moench) is a drought-tolerant cereal crop belonging to the Poaceae family. It is largely cultivated in Africa, Asia, and parts of America. Sorghum serves as a staple food for many people and is a major resource for animal feed and raw material for industries (Dicko et al., 2006). Sorghum's adaptability in arid and semi-arid areas, combined with its high biomass production, makes it a valuable crop for both food security and industrial applications.

Sorghum grains are abundant in carbohydrates (about 70–75%), with average quantities of protein (8–12%), and minute amounts of lipids (2–5%) as reported by Awika et al., (2004). The grains also contain vital quantities of dietary fibres and biologically active substances such as tannins, phenolic acids, and flavonoids. These compounds have demonstrated potential health impacts including antioxidant, anti-inflammatory, and anti-cancer activities (Dykes et al., 2006).

Sorghum is a vital primary material for bioethanol synthesis. Both sweet and grain sorghum types are utilized for this purpose. Sweet sorghum contains more sugar in its stalk. This high sugar content can undergo direct fermentation, but grain sorghum is rich in starch, which goes through saccharification prior to fermentation (Reddy et al., 2005). Also, the emergence of improved biofuels has increased curiosity in agricultural waste from sorghum as a potential raw material for biofuel synthesis.

Sorghum is largely utilized in producing local alcoholic drinks in Africa and Asia. It is used for the production of beers, pito, spirits, and traditionally preserved foods. In food industries, sorghum powder is used for making products such as biscuits, snacks, and porridge which are free from gluten (Taylor et al., 2006).

The fibrous remnants from the stalks and leaves of sorghum have potential in the paper and packaging industry. These cellulose-rich materials can be made into cardboard, paper and biopackaging. This decreases dependence on raw materials that are of wood origin (Gnansounou et al., 2005). Sorghum processing in industries produces a variety of byproducts, which can be refined for economic and environmental importance. The complete usage of sorghum and its byproducts aid the goals of the circular bioeconomy. It decreases waste, supports renewable energy, and provides income opportunities for brewers. Furthermore, the processing of by-products adds value to sorghum production chains and decreases the carbon footprint of brewing industries (Zegada-Lizarazu et al., 2012).

Cell walls of sorghum grains are made of primarily; non-starch polysaccharides, such as arabinoxylans, cellulose, and β-glucans. Arabinoxylans. These are abundant in hemicelluloses; and lignin are also found, particularly in the pericarp and testa layers, aiding the grain's hardness and resistance to digestion (Saulnier et al., 2007). Also, phenolic acids such as ferulic and p-coumaric acids present in the cell wall of the grain; further cross-link its wall components, making the cell wall resistant to degradation (Dykes et al., 2006).

Malting activates the enzymatic hydrolysis of complex carbohydrates. During germination, hydrolytic enzymes such as endo-β-1, 4-xylanase, arabinofuranosidase, and β-glucanase are produced. These enzymes degrade arabinoxylans and β-glucans. This loosens the cell wall matrix and increases porosity (Duodu et al., 2003). This breakdown makes the starch and protein bodies available during continued processing, such as mashing in brewing or cooking.

Malting enhances the functional properties of sorghum powder, such as water absorption capacity, bulk density, and swelling power, which are affected by changes in the cell wall. These modifications are vital in the production of weaning foods, breakfast cereals, and snacks (Obilana et al., 2003).

Malting lowers the amount of insoluble fibre by breaking non-starch polysaccharides, thus enhancing the nutritional value and bioavailability of nutrients. The degradation of cross-linked phenolic acids and lignin also helps in softening the cell walls. This makes the grain

more digestible (Taylor et al., 2001). Moreso, tannins and phytic acid, known anti-nutritional factors, are drastically decreased during malting.

2.3 Microcrystalline Cellulose Extraction Methods and Process

There are diverse protocols for extracting MCC from different sources. For example, the acid hydrolysis, which involves the treatment of cellulose with strong acids, such as sulphuric acid or hydrochloric acid, to break down the cellulose fibres into smaller MCC particles (Agboeze et al., 2022). The resulting MCC is washed and purified to eliminate acid further to make the product neutral (Agboeze et al., 2022). Enzymatic hydrolysis of MCC employs cellulase to break down cellulose into MCC (Saini et al., 2016). This extraction procedure is environmentally; friendly compared to the acid hydrolysis method (Agboeze et al., 2022). However, it is slower and more expensive (Shi et al., 2018). Methods such as high-pressure homogenization or micro-fluidization; can also be employed to extract MCC from cellulose sources (Agboeze et al., 2022). This mechanical procedure occurs via the breakage of cellulose fibres into smaller particles (Agboeze et al., 2022).

According to Avbunudiogba et al (2022), steam procedure used for MCC extraction exposes cellulose fibre to an elevated pressure steam. This step rapidly breaks the cellulose structure to produce refined MCC. Nazir et al (2023), successfully extracted MCC from Walnut, Almond and Apricot Stone Shells. In that work, they listed the importance of monitoring extraction parameters to optimize the extraction process (Nazir et al., 2023). The study also showed that renewable agricultural biomasses viz Walnut, Almond and

Apricot stone shells can be employed as an alternative raw material for the extraction of MCC by methods such as pre-treatment, using alkali bleach followed by acid-hydrolysis procedure (Nazir et al., 2023).

The MCC extraction undergoes a series of steps to extract and purify cellulose. Then, it is further processed into MCC. The MCC produced is suitable for a wide range of applications across different industries (Agboeze et al., 2022). The extraction processes include pulping into fibres or pulp, purification, hydrolysis and crystallization (Agboeze et al., 2022).

Breaking down raw cellulose-containing materials is typically done through mechanical or chemical processes to separate cellulose from lignin and other components (Nazir et al., 2023). Pulping is done to obtain a pulp that contains cellulose as a primary component for further processing into MCC (Nazir et al., 2023).

The purification step involves refining the pulp obtained via pulping to remove impurities such as lignin, hemicellulose, and other non-cellulosic materials (Nazir et al., 2023). This step is crucial for improving the quality and the characteristics of the cellulose used for MCC production (Agboeze et al., 2022). Purification ensures that the cellulose used for MCC extraction is of high purity in order to obtain good performance and consistent product quality in pharmaceutical and other industrial applications (Nazir et al., 2023).
Initially, an alkaline pretreatment process is employed to remove lignin and hemicellulose from the material to obtain a purified cellulose fraction. Sodium hydroxide (NaOH) is

commonly used in this step to optimize and reduce chemical consumption to achieve a high yield of cellulose (Nazir et al., 2023).

In the hydrolysis process, the cellulose molecules are broken down into smaller units, such as glucose by reacting the cellulose with water, acids or enzymes (Nazir et al., 2023). In the context of microcrystalline cellulose extraction, acid hydrolysis is commonly used to break down cellulose chains into smaller crystalline segments (Nazir et al., 2023). The purpose of hydrolysis in MCC production is to reduce the size of cellulose crystallites, which enhances their functionality and improves the flow properties, compressibility, and binding characteristics of MCC in pharmaceutical tablet formulations (Siro al, 2010).

Chemical hydrolysis is the predominant technique for isolating MCC. This process generally involves the use of strong mineral acids like hydrochloric acid (HCl) or sulphuric acid (H_2SO_4) to break down the amorphous regions of the cellulose to form microcrystals (Agboeze et al., 2022).

This method involves two main stages, viz acid Hydrolysis, where cellulose is treated with a concentrated acid, HCl or H_2SO_4. These acid target and then hydrolyzes the amorphous sections of the cellulose (Haque et al., 2015). The hydrolysis step is followed by washing to eliminate any remaining acid and dissolved sugars, and then neutralized to quench the reaction (Huang et al., 2020).

Enzymatic hydrolysis has emerged as a promising, eco-friendly alternative to acid hydrolysis (Kian et al., 2020). Enzymes such as cellulases, xylanases, and lignases selectively degrade non-cellulosic materials (Agboeze et al., 2022). This offer results in a more sustainable and environmentally friendly method for MCC production. (Tachoun et al., 2019). This approach is beneficial for agricultural residues in which cellulose is entangled with lignin and hemicellulose (Ul-Islam et al., 2012).

Recent innovations in MCC extraction focus on the use of non-chemical techniques such as steam explosion, microwave-assisted extraction, and supercritical fluid processing (Zhang et al., 2019). These innovative methods facilitate efficient cellulose extraction without the need for chemicals and thus provide a more environmentally friendly alternative pathway to MCC production (Zeleke et al., 2022).

The crystallization step in MCC extraction involves the formation of microcrystalline structures from the hydrolyzed cellulose (Zeni et al., 2016). After hydrolysis step, the cellulose becomes suspended in the solvent used. These suspended celluloses are then crystallized under controlled conditions such as temperature and pressure, to aid the formation of small, uniform and crystalline particles (Agboeze et al., 2022). The purpose of crystallization is to produce microcrystalline cellulose, which has specific particle size, shape, and crystallinity characteristics (Kunusa et al., 2018).

2.4 Characterization of Microcrystalline Cellulose

Characterization of MCC is very crucial for understanding its physical, chemical, and mechanical properties (Agboeze et al., 2022). Belali et al., (2019), investigated the effect of MCC particle size on pharmaceutical tablet compaction properties. These researchers found that a smaller MCC particle size resulted in higher tablet strength and reduced disintegration time.

MCC characterization involves a set of analytical protocols to explain its chemical structure, crystalline nature, morphology, and solubility behaviour: FTIR analysis is used to verify the removal of amorphous components of cellulose such as lignin and hemicellulose and to confirm the retention of native cellulose functional groups. For example, the absence of peaks at 1733 cm^{-1} aligned to lignin/hemicellulose C=O stretching indicates a highly purified MCC (Udo et al., 2020). The occurrence of peaks at hydroxyl (OH), C–H, and glycosidic (C–O–C) vibrations further confirms the cellulose structure as reported in a study by Tachoun et al., (2019).

XRD is utilized to evaluate the crystallinity of the extracted MCC. Specific diffraction peaks close to 15° and 22° (2θ) conform to cellulose I polymorphs, and the crystallinity index (CrI) is determined using the Segal protocol, revealing a clear understanding of the degree of crystallinity of the MCC structure (Asif et al., 2022).

SEM demonstrates the morphology and particle properties of MCC. MCC usually occurs as microfibrils or granular crystals, based on the material origin. For instance, MCC

produced from roselle fibres showed rough surface morphology and little aggregation (Uzondu et al.,2020).

Protocols such as Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) also evaluate thermal stability and moisture content of the extracted MCC. TGA mostly shows initial weight loss near 100 °C corresponding to moisture and main decomposition at a temperature range of 250–330 °C for the synthesized MCC (Uzondu et al., 2020).

The flow properties of MCC are largely based on its moisture content, shape, particle size, and surface texture. The poor flowability of MCC is generally due to the irregular and porous nature of the particles, while more granular MCC powders contribute to excellent flowability. Smaller particle sizes have greater interparticle forces, which aid them exhibiting more cohesiveness (Okoye et al., 2015).

Many studies have assessed MCC using standard flow indicators like Carr's Compressibility Index (CI), crystallinity index (CrI) and Hausner Ratio (HR). In a report by Aulton et al., (2013), a Carr's index ranging from 5–15% demonstrates excellent flow, whilst values greater than 25% indicate poor flow. MCC generally shows CI and HR values suggesting moderate to passable flow, depending on the raw material source and processing protocol employed. For example, native MCC usually have CI values falling between 15–23% and HR values in the range of 1.2–1.4 as indicated in a study by Uzondu et al., (2022).

MCC's flowability is closely attributed to its tapped and bulk densities. Low bulk density of MCC between 0.2–0.4 g/cm³ and high packing ability usually limit its flow. These characteristics can be enhanced via particle engineering protocols like co-processing or granule formation. Co-processed MCC demonstrates improved flow because of the particle size increased and decrease cohesiveness (Rojas et al., 2012).

Angle of repose of MCC is an important index. MCC normally demonstrates an angle of repose of 30 - 40°, showing fair flow properties (Alebiowu et al., 2003). However, enhancements like spray drying or combining with glidants such as colloidal silicon dioxide have been demonstrated to decrease the angle of repose and improve flowability.

CHAPTER THREE

METHODOLOGY

The chapter outlines materials and chemicals used, detailed step-by-step experimental protocols for the extraction of α-cellulose and its conversion to MCC. It also presents information the analytical techniques applied in characterizing the extracted MCC and its physicochemical properties.

3.1 Sampling Area

The malted sorghum mash sample for the study was picked from Amanten, a rural farming community located in the Atebubu-Amantin Municipality in the Bono East Region of Ghana. Geographically, Amanten lies within the forest-savanna transitional zone. The area is characterized by flat to moderately undulating terrain, with a mixed vegetation of grasses and scattered trees. The Amanten community experiences a tropical climate with distinct wet and dry seasons. Annually, the average rainfall ranges between 1,000 and 1,200 mm. This range of rainfall supports seasonal crop farming (Fuseini et al., 2024).

Amanten is primarily an agrarian community and the majority of the residents engaged in crops cultivation. For example, examples of crops cultivated in the Amanten community include but are not limited to maize, yams, beans, cassava, and sorghum among others. Sorghum malting and traditional brewing are common in the area. In the Amanten community, malted sorghum mash is readily available as a by-product from small-scale brewing operations (Ansah et al., 2023). Therefore, the community is strategic and a

practical choice for sourcing malted sorghum mash, which is needed as a raw material for this work.

The Amanten community has basic infrastructure which includes but not limited to market, schools, and health facilities. Transportation in and around Amanten is by unpaved roads. Though, the roads are accessible, they become challenging to access during the rainy season (Ansah et al., 2023).

3.2 Sampling of Malted Sorghum Mash

Sampling was carried out from multiple household-based processing points where traditional sorghum malting and fermentation are routinely practiced, ensuring the material collected was representative of typical local production processes. This setting not only provided access to fresh and abundant malted sorghum mash but also reflected real-world processing conditions relevant to exploring the feasibility of MCC production from agro-industrial residues in rural Ghanaian contexts.

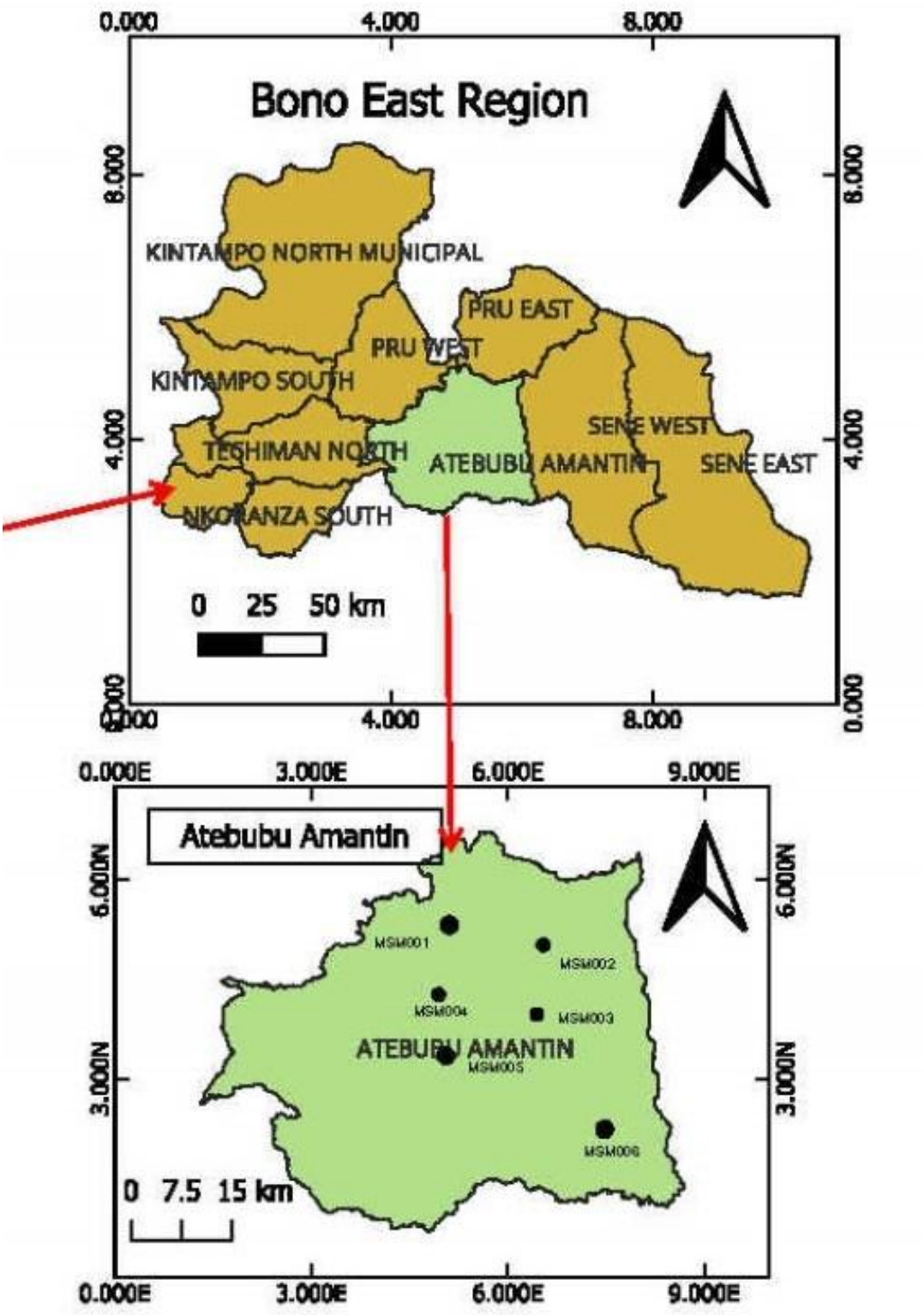

Figure 3.1: The map showing the sampling area

3.3 Sample Preparation

The malted sorghum mash was collected from multiple sorghum processing points in the Amanten community. They were washed thoroughly with distilled water. The washing was done to remove dust, fungus, water-soluble components and other foreign materials from the malted sorghum mash, which is hereafter referred to as the sample. The sample was air-dried for 72 hours and then pulverized into powder with a mortar and pestle. Here, 100g each of the dried malted sorghum mash was placed into the mortar to allow space for thorough grinding. It was then hand-pressed and crushed with the pestle in a downward motion to break large particles. The hand pressing and crushing continued with firm circular grinding motions to reduce the particles to a fine powder. Consistent pressure was applied and pounding was avoided to reduce sample loss. The ground mash was sieved by passing it through a 250 μm sieve to achieve uniform particle size. Any retained coarse particles were reground and sieved. The pulverized malted sorghum mash was transferred into a clean, labelled container. It was then stored in an airtight bag to avoid moisture absorption.

3.3.1 Chemicals Employed

All chemicals used were of analytical grade. They included; Sodium hydroxide (BDH, England), 4% w/v sodium hypochlorite (British Drug House, Poole, England), hydrogen peroxide and hydrochloric acid (Fisons, UK). The reagents used were; 3.0M HCl and 40% (v/v) hydrogen peroxide.

3.3.2 Equipment Employed

The equipment used were; pH meter Hanna (HI831, USA), electronic balance (XLB1054, China), freeze drying machine (Alpha 1-4LSC, Germany), water bath (888392,Egland), oven (GP/50/CLAD/250/HYD, Egland), Scanning Electron Microscope JOEL analytical Microscope(TSM6380, USA), X-Ray Diffractometer (D2 PHASER), Perkin-Elmer FT-IR spectrometer (Spectrum Two, Japan), TGA-DSC thermal analyzer (SDT Q600 V20.9 Build 20, USA).

3.3.3 Preparation of Solutions

3.3.3.1 Preparation of 4% (w/v) NaOH aqueous solution

Forty grams of solid NaOH pellets were weighed into a 100cm^3 clean beaker.30 cm^3 of distilled water was added. The distilled water and NaOH mixture were stirred continuously to ensure complete dissolution of the NaOH pellets. The resulting solution was then transferred to a 1 L volumetric flask. The beaker and the stirring rod were rinsed three times each with some distilled water and added to the contents of 1 L. More distilled water was added to the contents until it reached the 1 L mark on the neck of the flask. The flask was corked, labelled and stored for use.

3.3.3.2 Preparation of 3.0M HCl (37% purity) aqueous solution

In a fume hood, 250 mL of 37% HCl was put into a 1 L volumetric flask previously filled 500 mL of distilled water. The measuring cylinder used to deliver 250 mL of the 37% HCl was rinsed thrice each with 50 mL distilled water and then added to the solution in the 1 L volumetric flask on each rinsing. The mixture in the flask was swirled gently for 2 minutes.

More distilled water was added to reach the 1 L mark on the neck of the flask. The flask was then corked, labelled and stored for use.

3.3.3.3 Preparation of 5.5% Sodium hypochlorite solution

In a fume chamber, 458 mL of 12% sodium hypochlorite solution was put into 1 L volumetric flask previously filled with 200 mL distilled water. The measuring cylinder used to transfer the 12% sodium hypochlorite solution was rinsed thrice each with 50 mL distilled water and then added to the contents of 1 L volumetric flask after each rinse. The mixture in the flask was swirled gently for 2 minutes. More distilled water was added to reach the 1 L mark on the neck of the flask. The flask was then corked, labelled and stored in a dark place away from light for use.

3.3.3.4 Preparation of 40% (v/v) H_2O_2 solution

In a fume chamber, 400 mL of 40% hydrogen peroxide stock solution was put into 500 mL volumetric flask previously filled with 50 mL of distilled water. The measuring cylinder used to transfer the 40% hydrogen peroxide stock solution was rinsed thrice each with 10 mL of distilled water and then added to the contents of 500 L volumetric flask after each rinse. The flask's content was made to reach the 500 L mark on its neck by adding distilled water. The flask was then corked, labelled and then stored in an opaque container to avoid decomposition by light for use.

3.3.4 Extraction of α-Cellulose from Malted Sorghum Mash

The method used by Nazir et al (2023) to extract α-cellulose was adopted, slightly modified and used in this current work. One hundred and twenty-five grams of pulverised malted sorghum mash powder was put into a 1000 mL glass beaker, and 400 mL of 4% (w/v) previously prepared sodium hydroxide solution was added. The mixture was stirred thoroughly for 3 minutes and then kept in a water bath at 80°C for two hours. The cellulose pulp was filtered through previously sterilized calico cloth. The resulting cellulose pulp was thoroughly washed with copious amount of tap water, and pH of the filtrate was determined using a previously calibrated pH meter (HI8341, USA). The washing was repeated until the pH of the filtrate became neutral. The residual cellulose pulp was bleach with 250mL of 5.5% (v/v) sodium hypochlorite solution previously prepared and kept at 60˚C for 2 hours in a water bath. The resultant slurry was filtered through a sterilized calico cloth and further washed thoroughly with distilled water until a neutral pH was obtained. This α-cellulose extraction was repeated under the same experimental conditions until a milky white α-cellulose crystalline were obtained.

The α-cellulose crystals were bleached with 250 mL of 40 %(v/v) hydrogen peroxide solution for 3 hours in a water bath at 50°C. The bleached α-cellulose crystals were filtered under gravity and then washed multiple times each with a copious amount of distilled water until neutral α-cellulose crystals were obtained. Remaining water in the extracted α-cellulose crystals was manually removed by placing the crystals into a sterilized calico cloth and the water was squeezed out to obtain small lumps of α-cellulose crystals. The

small crystal lumps obtained were used for MCC extraction. The percentage α-cellulose crystals produced was calculated using the equation below.

$$\% \text{ Yield of } \alpha\text{-cellulose} = \frac{m_{\alpha c}}{m_s} \times 100\% \qquad 3$$

Where; $m_{\alpha c}$ and m_s Represent the mass of α-cellulose and the sample, respectively.

3.3.5 Microcrystalline Cellulose Extraction (MCC)

The method of microcrystalline cellulose extraction employed by Nazir et al. (2023) was adopted and slightly modified, and used in this work. One hundred and fifty grams of previously extracted α-cellulose was put into a glass container, 400 mL of 3.0M hydrochloric acid was added and samples allowed to hydrolyse at a 108°C for 1 hour. The resultant hot acid mixture was poured into a 1 L of distilled water, and the mixture was vigorously stirred with a stirring rod for 10 minutes, and then allowed to stand overnight at room temperature.

$$(C_6H_{10}O_5)_n + HCl_{(aq)} \xrightarrow[108^{\circ}C]{Limited\ time} \text{MCC} + \text{Hydrolyzed fragments} \qquad 4$$

The MCC produced was filtered, and the residue was washed thoroughly with distilled water at room temperature. The washing procedure was repeated several times until the water was neutral to litmus paper. The MCC extracted was transferred into a 600 mL plastic container, 400 mL distilled water added, the lid of the container was closed, sample was labelled (MSM-MCC), freeze-dried and then stored in a previously cleaned air-tight container for instrumental analysis.

3.3.6 Determination of Physicochemical Properties of Extracted MCC

The organoleptic characteristics, viz taste, odour and colour, identification of organic impurities with phloroglucinol, starch and dextrin, solubility and total ash assessments were done in accordance with the British Pharmacopoeia (2023) specifications.

3.3.7 Characterization of Extracted MCC

To establish the identity of the extract, selected chemical tests were performed on the extract. These were chemical tests to determine the occurrence of lignin, starch and sugar.

3.3.7.1 Test to determine presence of lignin in the MCC extract

One gram of the extracted MCC was put into a test tube, 10 mL of 1% phloroglucinol in ethanol was added to the content in the test tube and 2 mL of 37% (w/v) concentrated hydrochloric acid was added. The resultant mixture is mixed gently and then allow to stand for 5 minutes. Red or purple colouration shows the presence of lignin, while no change indicates the absence of lignin.

3.3.7.2 Test to determine the presence of starch in the MCC extracted

Half gram (0.5g) of the extracted MCC powder is dispersed into a boiling tube, previously filled with 5 mL of distilled water. The resulting mixture is boiled gently for 2 minutes and then allowed to cool to room temperature. This was followed by the addition of two drops of iodine solution, and the extract was observed for any change in colour and recorded. Blue or purple colouration indicates the presence of starch, while no change indicates the absence of starch.

3.3.7.3 Test for sugar presence in MCC extract

The standard British Pharmacopoeia (BP) method for testing free reducing sugars was adopted and used. One gram of the MCC extract was put into a 100 mL beaker, 10 mL of distilled water was added and the mixture was stirred thoroughly using a glass rod. The resultant solution was heated gently for 5 minutes to suspend the cellulose and then leach out soluble impurities which may include reducing sugars. The mixture was allowed to cool to room temperature and then filtered using Whatman (No. 1) filter paper to obtain a clear filtrate.

In a clean test tube, 1 mL of Fehling's A solution and 1 mL of Fehling's B solution were added. Two millilitres of the clear filtrate solution obtained from the MCC extract were added to the mixed Fehling's reagent. The resultant solution mixture of the MCC in the test tube and the Fehling's reagent was gently heated in a Bunsen flame for 10 minutes and any colour change was observed. A brick-red or orange colouration indicates the presence of reducing sugars, while no change indicates the absence of reducing sugars.

3.3.8 Scanning Electron Microscopy (SEM) Analysis of MCC for Surface Characteristics of MCC

Powder of the MCC extract was dried completely at 60°C in an oven for 24 hours to remove residual water molecules. The dry MCC powder was mounted onto the aluminum stubs of SEM (Joel 6310) by sprinkling a small amount of MCC powder onto the aluminum stubs. The sputter coater of the SEM was used to apply 10 nm thin conductive coating of the MCC extract to prevent charging and improve the final image. The sputter-coated

sample was inserted into SEM chamber, and the image taken under vacuum at 15 kV accelerating voltage.

3.3.9 Infrared Spectroscopy (FTIR) for Functional Group on the MCC

MCC powder was thoroughly dried at 60 °C in an oven for 24 hours to remove moisture, was ground into a fine powder using mortar and pestle. Two milligrams of the ground MCC were mixed with 100 mg of dry KBr, and the mixture was further ground thoroughly to ensure homogeneity. The resulting mixture of MCC and KBr was pressed into a transparent disc using the press. The disc was inserted into the FTIR instrument and the spectra were recorded using 4,000 to 400cm^{-1}. Portions of the electromagnetic radiation were recorded.

3.4 X-ray Diffraction (XRD) Analysis of MCC Powder for Structural Morphology

X-ray diffraction analysis was performed using an x-ray diffractometer (Philips Xpert MPD). The MCC extract was dried to remove moisture and then ground to obtained uniform fine powder. The ground powder was then loaded onto a background sample holder and pressed into a smooth surface, inserted into the XRD machine, and then scanned from 5 to 40° 2θ to obtain the diffraction pattern of the MCC extract. The crystallinity index (CrI) was calculated using Equation (5).

$$\text{CrI} = \frac{I_{002} - I_{am}}{I_{002}} \qquad 5$$

Where I_{002} represents the maximum intensity of the principal peak lattice diffraction, and I_{am} represents the intensity diffraction associated with the amorphous cellulose.

3.4.1 Thermal Analysis of MCC Powder

The thermal characteristics of the extracted microcrystalline cellulose (MCC) samples were investigated by thermogravimetric analysis (TGA) and DSC simultaneously on thermal analyser. Samples ranging from 6 mg to 10mg were used. Each sample was heated from 30°C temperature to 500°C at a rate of 5°C/min under nitrogen.

3.4.2 Determination of Percentage Yield of MCC

The percentage yield of the extracted MCC was calculated using the equation

$$\%\text{yield} = \frac{y}{x} \times 100, \qquad 6$$

Where; y is the mass (mg) of the extracted MCC and x (mg) is the mass of the extracted alpha cellulose.

3.4.3 pH Determination of MCC

The pH meter was calibrated using a three-point calibration method with pH buffers (7.0, 4.01 and 10.0) as follows; the pH meter was turn on and allowed to warm up for 5 minutes. Its electrode was rinsed with distilled water and gently blotted dry with a lint-free tissue. 50 mL of pH 7.0 buffer was then put into a clean beaker. The electrode of the pH meter was immersed-fully in the solution, ensuring the junction and bulb were submerged. It was stirred gently with the electrode and the reading was allowed to stabilise. Once stable, pH 7.0 was confirmed on the meter and then calibrated by selecting calibrate button on the pH meter. The electrode was rinsed with distilled water and blotted dry. The procedure above was repeated under the same experimental conditions for pH buffers 10.0 and 4.0 respectively. Ten grams of MCC powder was shaken with 200 mL of distilled water for

5minutes. The probe of the pH meter was inserted into the supernatant liquid and the pH was recorded using a pH meter (Hanna HI83141). The pH was recorded when the pH meter readings did not vary more than 0.2.

3.4.4 Total Ash Content Determination of MCC

The measuring MCC residue estimated ash content left after combustion in a furnace (Agboeze et al., 2022). Three grams of MCC powder were put into a previously weighed crucible using an electronic balance. The mass of the crucible and the residue were also determined and the total ash content computed using the expression below

$$\text{Ash content (\%)} = \frac{W_1 - W_2}{W_1} \times 100\% \qquad 7$$

Where W_1 is the weight of the sample prior to the combustion and W_2 is the weight after combustion.

3.4.5 **M**CC Particle Size Analysis

Ten grams of the extracted MCC from malted sorghum mash was carefully poured onto the center of a 50µm sieve. The sieve was then manually shaken in a side-to-side motion for 10 minutes.

3.4.6 Flow Properties of Extracted MCC

Flow properties refer to the behaviour of the MCC powder particles when subjected to gravitational or mechanical forces. These properties were evaluated to evaluate the suitability of MCC for use in solid dosage forms such as pills and capsules. The flow properties of the extracted MCC were evaluated via various empirical parameters such as

bulk density, tapped density, Carr's Index, Hausner Ratio, and angle of repose based on standard procedures outlined below.

3.4.6.1 Angle of Repose of extracted MCC

The angle of repose refers to the maximum angle between the surface of a heap of the MCC powder and the horizontal plane. It indicates the internal friction between particles and is a quick estimate of flow potential (Agboeze et al., 2022).

The static angle of repose was measured according to the fixed funnel and free-standing cone method reported by Agboeze et al., (2022). A funnel was clamped with its tip 2 cm above a sheet of graph paper placed on a flat horizontal surface. The powders were carefully poured through the funnel until the apex of the cone just reached the tip of the funnel. The mean diameter of the base of the powder cone, which were determined by averaging the two diameters of the base of the cone which was measured in two perpendicular directions using a ruler. The tangent of the angle of repose is calculated using the Equation:

$$\text{Tan } (+\Theta) = \frac{h}{r} \qquad 8$$

Where h (cm) is the height of the heap of powder, Θ is the angle of repose (degrees) and r (cm) is the radius of the base of the heap of powder.

3.4.6.2 Bulk and Tap Densities determination of extracted MCC

For the determination of the tapped and bulk densities, the methods reported by Roshni et al., (2017) were adopted with slight modification. Thirty grams of the extracted MCC

powder was placed into a 300 mL previously clean, dry measuring cylinder and the volume (Vo), occupied by the samples without tapping was recorded. After100 taps, the volume (V_{100}) occupied was also recorded. The bulk and tap densities were calculated as the ratio of weight to volume (V_{100} and V_0) respectively. The Carr's index and Hausner's ratio were determined from the values of the bulk and tapped densities results obtained above. The bulk density D_{bulk} and the tap density D_{tap} were calculated using the equations below;

$$D_{tap} = \frac{W}{V_{100}} \quad (9)$$

$$D_{bulk} = \frac{W}{V_0} \quad (10)$$

3.4.6.3 Hydration Capacity of extracted MCC

One-gram samples were placed in each of the 4 different 30 mL plastic centrifuge tubes, 20mL of distilled water was added, and then the tubes were corked. The contents were manually mixed for 2 minutes, and the mixture allowed to stand for 10 minutes. After 10 minutes, the mixture was immediately centrifuged at 1000 rpm for 10 minutes using a bench centrifuge (TDL4 Tabletop low speed centrifuge, Gallen Kamp, England). The supernatant was decanted, and the sediments were weighed. The hydration capacity of the extracted MCC was taken as the ratio of the weight of the sediment to the dry sample weight as indicated in the equation below.

$$H_c = \frac{W_s}{W_{ds}} \quad (11)$$

Where; H_c, W_s (g) and W_{ds} (g) represent hydration capacity, weight sediment and dry sample weight respectively.

3.4.6.4 Swelling capacity of extracted MCC

The swelling capacity (S_c) of the powder MCC was estimated as described by Avbunudiogba et al. (2022) with slight modification. In this method, the tapped volume (V_x) occupied by 3.0 mg of the powder was noted, and the powder was dispersed in 85mL of distilled water, and the volume made up to 100 mL. After 24 hours of standing, the volume of the sediment (Vv) was measured. The swelling capacity was then computed as the ratio of the volume of sediment to the tap volume as shown in the equation below.

$$S_C = \frac{V_v}{V_x} \qquad 12$$

3.4.7 Moisture Absorption Capacity of Extracted MCC

One gram of the MCC extracted was accurately weighed and evenly distributed over the surface of a 90 mm tarred Petri dish. The sample was then put into a large desiccator filled with 50 mL of distilled water in its reservoir (RH = 100 %) at room temperature. The weight gained by the exposed samples over 5 days period was recorded and the amount of water absorbed was calculated from the weight differences using equation (10).

$$W_{wa} = \frac{w_1 - w_2}{w_1} \times 100\% \qquad 13$$

Where W_{wa} is the weight of the water absorbed, W_1 is the weight of the samples before exposure, and W_2 is the weight of the samples after exposure.

3.4.8 Loss of Weight on Drying

Three grams of the extracted MCC were put into a Petri dish and dried in an oven at 105°C until a constant weight was obtained. The percentage moisture content was determined using equation below.

$$\%\text{Loss of weight on drying} = \frac{W_i - W_f}{W_i} \times 100\% \qquad 14$$

Where:

W_i = Initial weight of the MSM-MCC sample before drying (g)

W_f = Final weight of the MSM-MCC sample after drying (g)

CHAPTER FOUR

RESEARCH FINDINGS AND DISCUSSIONS

This chapter presents the findings from the extraction of MCC and characterization experiments. It also interprets and discusses the results in light of existing literature, providing insights into the suitability and potential of malted sorghum mash as a cellulose source.

4.1 Physicochemical Properties

The malted sorghum mash experienced multistage pulverizing, which successfully delignified the material. This process produced a uniform α-cellulose pulp, signifying the method's efficacy in considerably removing lignin. From 125 grams of the MSM, a yield of 70% α-cellulose pulp was attained. The pH of the malted sorghum mash MCC was measured at 6.9, falling within the acceptable range of 5-7.5 (Agboeze et al., 2022). The physical and chemical properties of the MSM- MCC extracted by acid hydrolysis using $HCl_{(aq)}$ are shown in Table 4.1. The Organoleptic properties of the MSM-MCC produced is good as the extracted material is tasteless, odourless and almost white granular powder

Table 4.1: Physical, chemical and organoleptic properties of the extracted MSM-MCC.

Property	Observed value
Appearance	Granular
Colour	White
Odour	Odourless
Taste	Tasteless
pH	6.9
Sugar	Negative
Lignin	Negative
Starch	Negative
Ethanol	Insoluble
Distilled water	Insoluble
Dilute HCl	Insoluble
Acetone	Insoluble

4.1 SEM Analysis of MSM-MCC

The surface morphology of the extracted MSM-MCC was examined using scanning electron microscopy (SEM). It provides detailed images of the surface structure of the MCC produced. The SEM micrographs of the extracted MSM-MCC are shown in figure4.1.

Figure 4.1: SEM micrograph of the extracted MSM-MCC

The Scanning electron micrographs (Figure 4.1A and 4.1B) of the extracted MSM-MCC processed samples had outward projection of microfibrils. These microfibrils indicate that the cementitious lignin and hemicellulose constituents from the novel MSM sample were removed during synthesis. The length of the microfibrils ranged from 20 to 200µm

In the image shown in Figures 4.1A and 4.1B, the occurrence of the microfibrils shows that the microfibrils have helicoidal structured strands which align with the naturally ordered chains of cellulose. The structure of the MSM-MCC is a result of the organized chains of the pure native cellulose. It indicates the microcrystalline nature of the extracted product. The microfibrils also exhibit clumps which are spaghetti-like formations on the surface of the MSM-MCC. This spaghetti-like structure proved the crystalline nature of the product.

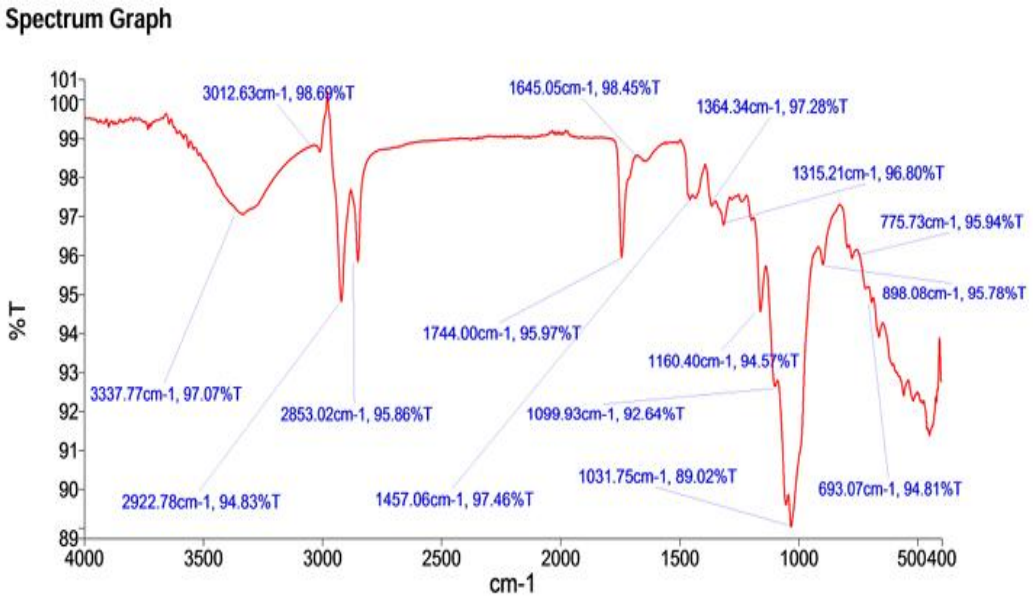

Figure 4.2: FTIR spectrum of extracted MSM-MCC

Molecular construction of the MSM-MCC was elucidated through FTIR results obtained within 4000 to 400 cm^{-1} wave length, similar to that reported by Li et al., (2019). The spectra obtained provided information on the functional groups present in the MSM-MCC material synthesized, and to some extent the composition of the synthesized material. The FTIR data of the MSM-MCC produced was used to determine the identity of the MSM-MCC has been indicated in a study by Agboeze et al.,(2022). The peak detected at 693.07 cm^{-1} indicates C-H bending vibrations in aromatics or ring deformation, signifying the occurrence of possible cellulose ring deformation or minor aromatic impurities in the MSM-MCC sample.

The peak at 775.73 cm^{-1} is due to ring deformation or skeletal vibrations of the pyranose ring, suggesting the integrity of β-D-glucopyranose ring in the cellulose structure while the peak at 898.08 cm^{-1} occurred due to C–H bending of the β-glycosidic linkage. This peak

indicates β-1,4-glycosidic linkages which confirms the presence of the cellulose structure in the synthesised MSM-MCC. The two peaks at 1031.75 cm^{-1} and 1099.93 cm^{-1} were due to C–O stretching in alcohols, ethers, or polysaccharides and C–O–C stretching in ether linkage respectively. The presence of these two peaks indicated the presence of cellulose in the sample. The peaks at 1160.40 cm^{-1} and 1315.21 cm^{-1} are suggested to have originated due to asymmetric C–O–C stretching in cellulose backbone in the sample. These C-O-C stretching peaks correspond well with those reported by Romruen et al., 2022) and indicate the presence of glycosidic linkages of cellulose in the MSM-MCC synthesised.

Furthermore, the peak witnessed at 1364.34 cm^{-1} was credited to the O–H bending, which is important for hydrogen bonding. The peak at 1645.05 cm^{-1} also showed H–O–H bending of water adsorbed, and it shows the presence of bound water found in hygroscopic MCC.

The peak at 1744.00 cm^{-1} indicated the stretching vibration of the C=O group, suggesting minor impurities or residual lignocellulosic components in the MSM-MCC extracted. Peak a 2853.02 and 2922.98 cm^{-1} were due to the C–H symmetric stretching in cellulose backbone and confirm aliphatic chains in the MSM-MCC synthesized. Peak 3012.63 cm^{-1} specifies the existence of =C–H stretching of minor unsaturation or contaminants and it indicates trace impurities or degradation in the MSM-MCC sample synthesized. Lastly, the peak at 3337.77 cm^{-1} can be ascribed to the O-H stretching vibration, signifying the manifestation of a broad peak typical of cellulose hydroxyl groups in the extracted MSM-MCC (Agboeze et al., 2022).

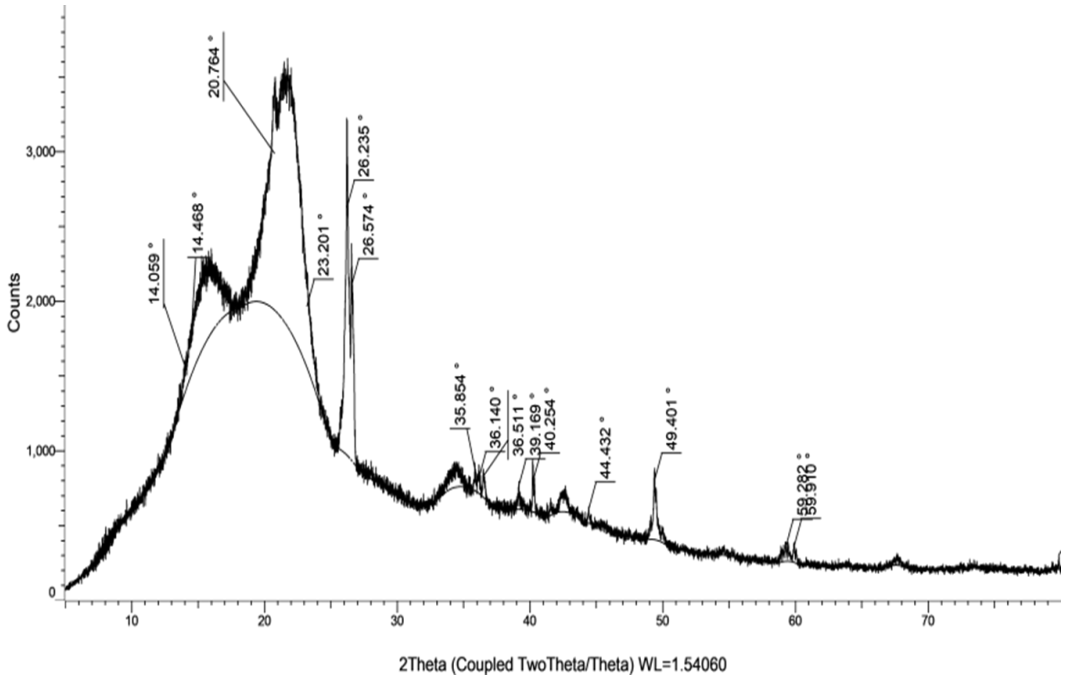

Figure 4.3: X-Ray Diffractogram of MSM-MCC extracted from MSM

The X-ray diffraction (XRD) analysis of the extracted MSM-MCC demonstrated multiple prominent peaks. These showed a semi-crystalline structure of the MSM-MCC synthesized. The main diffraction peaks were observed at 2θ values of 14.059°, 14.468°, 20.764°, 23.201°, 26.235°, and 26.574°, corresponding to interplanar spacing (d-spacing) values of 6.294 Å**,** 6.117 Å, 4.274 Å, 3.831 Å, 3.394 Å, and 3.352 Å, respectively. These peaks are consistent with the characteristic diffraction patterns of cellulose I, suggesting that the crystalline structure of the native cellulose was largely preserved during the extraction process.

The peak at 26.235°, with a d-spacing of 3.394 Å, recorded the highest net intensity of 1660.81, followed closely by the peaks at 26.574° (1170.36) and 20.764° (1048.54). These high-intensity reflections confirm the presence of highly ordered crystalline regions within

the cellulose matrix. The peak at 14.468°, with an intensity of 221.729, is often attributed to amorphous cellulose regions.

Additional peaks of lower intensity were observed at higher angles, notably at 35.854°**,** 36.140°, 36.511°, 39.169°, 40.254°, 44.432°, 49.401°, 59.282°, and 59.910°, with d-spacing values between 2.50 Å and 1.54 Å. These may correspond to secondary crystalline planes or minor impurities, but their lower intensities suggest limited influence on the bulk crystallinity. The presence of both sharp and broad peaks reflects the composite nature of MSM-MCC, comprising both crystalline and amorphous domains. This structural profile is favourable for applications requiring good compressibility, binding, and flow properties, especially in the pharmaceutical formulation of drugs.

The Segal method was employed to determine the Crystallinity Index (CrI), using equation (2). The intensity of the main crystalline peak (I_{002}) occurred at 26.2° (Figure 4.3) and corresponds to 1660.81 counts and the intensity of the amorphous background (I_{am}) occurred at 14.4° (Figure 3) with a value of 221.729 counts. Substituting these values into Equation (2), yielded a crystallinity index of 86.63%. This shows that the extracted MSM-MCC possesses a high degree of crystallinity. This indicates that the acid hydrolysis process effectively removed most amorphous regions, enriching the crystalline cellulose content. High crystallinity is generally associated with enhanced mechanical strength and reduced moisture absorption, making it suitable for pharmaceutical and industrial applications.

4.2 Thermostability of Extracted MSM-MCC

The TGA-DSC analysis was conducted to assess both the thermal stability and phase transition behaviour of MSM-MCC. TGA was used to measure the percentage weight loss of the sample as a function of temperature, while DSC measured the associated heat flow, revealing endothermic and exothermic events. Figure 3 shows the overlaid TGA-DSC curves for MSM-MCC across a controlled heating range. Each curve reflects a thermal event that corresponds to either a physical or chemical transformation within the MSM-MCC material.

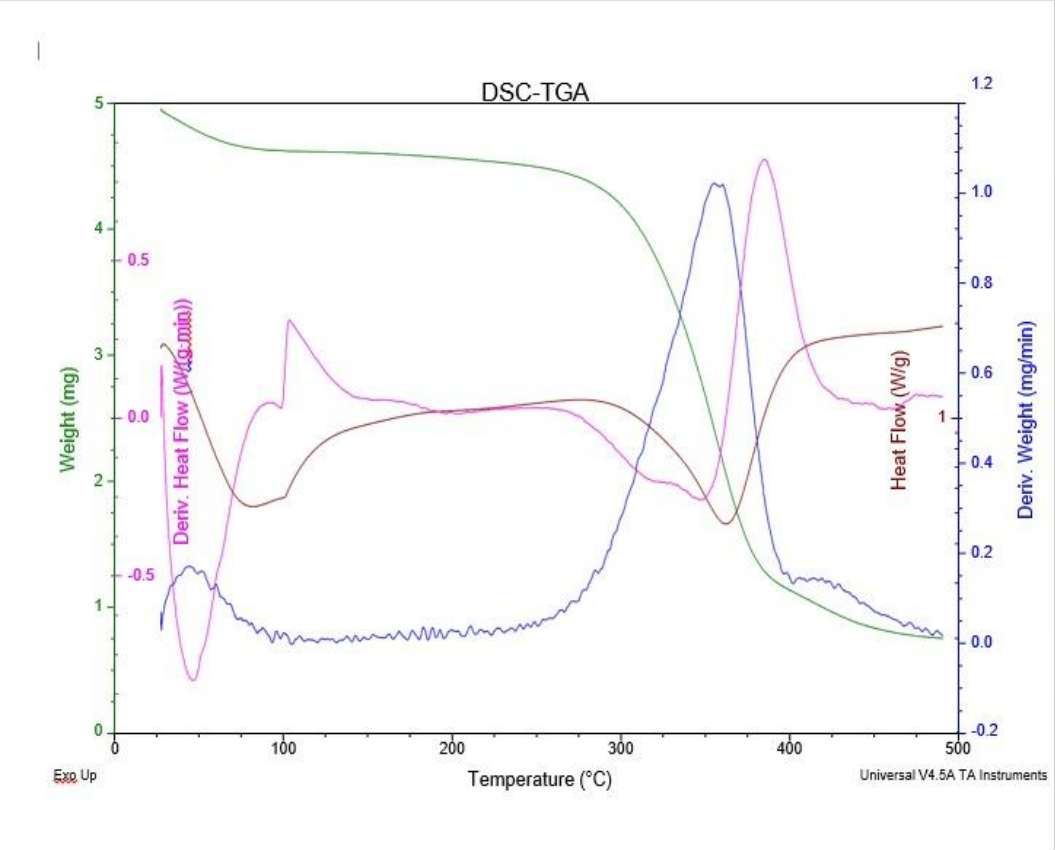

Figure 4.4: TGA-DSC curves showing weight loss and heat flow of MSM-MCC

Figure 3 showed the TGA curve of MSM-MCC synthesized. The MSM-MCC showed a drastic weight loss within the temperature range of 30-100°C.This suggests the presence of moisture content in the MSM-MCC sample. The MSM-MCC produced has hydrophilic character and the water molecules absorbed were lost upon heating. A moderate loss of weight occurred at a temperature ranging from 100 to 275.5°C. The mass loss was 0.075 mg and it indicates that MSM-MCC produced has good thermal stability in the 100–275.5°C temperature range. This shows the presence of cellulose in the MSM-MCC as reported by Karna et' al., (2022).

However, a rapid weight loss occurred at temperature ranging from 275.5–387.5°C (4.675 - 1.25 mg, figure 3) suggests a more significant thermal degradation of the MSM-MCC produced. This corresponded to the temperature decomposition of cellulose which substantial breakdown of polymer cellulose occur as reported by Karna et' al., (2022). Minimal weight loss occurs in the temperature range from 387.5–477°C. This indicates major degradation reactions occurred in the MSM-MCC sample before this temperature range. The residue left at higher temperatures were mostly charr and the observation aligned well with that made by Fouad et' al., (2020).

The TGA profile (Figure 3) indicates that the MCC produced from malted sorghum mash has a comparable degradation pattern to that observed by Fouad et' al., (2020), though they have different thermal stability. The TGA results (Figure3) highlighted the thermal behaviour of the extracted MSM-MCC. This Figure indicated suitability of the MSM-MCC produced in this work to be good for various applications, particularly in

pharmaceuticals and food. This observation also aligned well with that made by Tessema et al., (2023).

The MSM-MCC produced appear to be suitable for various applications, assessment of its purity, consistency and mechanical properties determine its effectiveness when compared to commercially available MCC.

A weight loss of 87.46% was obtained from the TGA of extracted MSM-MCC. This significant weight loss could be due to decomposition of cellulose structure, loss of moisture and loss of its volatile components. This weight loss suggests that a substantial portion of the MSM-MCC sample was thermally degraded. Though loss in weight of MCC due to moisture and decomposition can vary, such a high percentage loss may have occurred due to the presence of residual components during extraction process. (Tessema et al., 2023).

The high weight loss observed in the SD-MCC may impact its suitability as a pharmaceutical excipient. It could indicate that the extracted cellulose has a significant amount of non-cellulosic materials or lower crystallinity, which may affect properties like flowability, compressibility, and stability in formulations (Fouad et' al., 2020). Though MSM-MCC produced is suitable for use in various processes, its high percentage weight loss observed could impact its suitability as a pharmaceutical excipient. This huge loss could have occurred due to the presence of significant amount of non-cellulosic materials

or lower crystallinity and this may affect its flowability, compressibility and stability in pharmaceutical formulations as reported by Fouad et al., (2020).

The DSC thermogram (Figure3) provided further insights into the MSM-MCC material transitions. MSM-MCC sample in figure3 demonstrated an endothermic event in the temperature range of 50-100°C corresponding to water loss. The peak was broader and less intense, confirming its reduced moisture affinity. MSM-MCC showed another endothermic peak at the temperature ranging from 100 to 350°C. This peak indicates that the MSM-MCC synthesised is a crystalline material undergoing decomposition.

At 350-375 °C, the heat flow of the MSM-MCC drops, indicating a huge thermal event, due to the onset of cellulose degradation. This is common for cellulose as it begins to break down and decompose thermally. This peak at 350-375 °C also suggests a continued degradation and a possible release of volatile components as the cellulose undergoes further thermal breakdown. The heat flow values in the extracted MSM-MCC results suggest that the extracted MSM-MCC might contain a mix of amorphous and crystalline regions. In contrast, commercially available MCC usually presents a higher degree of crystallinity, which impact its thermal behaviour.

In conclusion, the DSC results of the extracted MSM-MCC indicate it has distinct thermal properties that reflect its composition and possible impurities. While it shows multiple thermal events, suggesting variability in crystallinity and potential moisture content,

pharmaceutical-grade MCC is characterized by greater thermal stability, higher crystallinity, and consistent thermal behaviour as reported by. Agboeze et al., (2022).

Table 4.2: Micromeritic properties of the MSM-MCC sample

Parameter	MSM-MCC
Bulk density (g/ml)	0.104
Tapped density (g/ml)	0.130
Carr's Index (%)	20
Hausner Index	1.25
Angle of repose	37
Swelling capacity	1.82
Moisture content (%)	5
Particle size(µm)	50.50
%Yield	60
Crystallinity Index (%)	86.63
Ash content (%)	0.6

The percentage ash content of the extracted MSM-MCC was found to be 0.6%. This indicates the absence of unwanted residues. Ash content indicates the amount of inorganic material present after the sample has been incinerated. In pharmaceuticals, low ash content is desirable as it suggests purity and minimal contamination (Agboeze et al., 2022). The percentage moisture content of the extracted MSM-MCC was found to be 5%. This is within acceptable limits as per pharmaceutical standards, ensuring stability and ease of handling. This showed that, the amount of water present in the MSM-MCC sample is low due to the stability of the MSM-MCC synthesised. A bulk density of 0.104 g/ml was obtained from the extracted MSM-MCC which suggests that the extracted MSM-MCC is

relatively loosely packed without external forces. This could affect flow properties and dosage uniformity of MSM-MCC as an excipient in pharmaceutical manufacturing.

The tapped density of the extracted MSM-MCC was found to be 0.130 g/mL, which is higher than the bulk density (0.104 g/mL). This indicates that the MSM-MCC sample compacts well under tapping. The MSM-MCC synthesized showed a minimal difference of 0.026 g/mL between its tapped and bulk densities. This indicates low compressibility and good flowability. This further suggest that, the MSM-MCC is efficiently packed in its bulk state and may be well-suited for applications such as direct compression in drugs formulations, where consistent flow and uniformity are important as indicated by Fouad et' al., (2020).

The Hausner index is calculated as the ratio of tapped density to bulk density and found to be 1.25 for the extracted MSM-MCC. This suggests that, the MSM-MCC produced has a fair to passable flow properties. Hausner index below 1.25 generally indicates good flowability, while values above 1.25 may suggest poor flow, impacting manufacturing processes like tableting or encapsulation.

Carr's index, also known as the compressibility index was calculated to be 20%, which indicates moderate compressibility of the MSM-MCC produced. Values below 15% typically denote good flowability, whereas values above 25% suggest poor flow properties as reported by Agboeze et al., (2022).

A crystallinity index of 86.63% was obtained from the extracted MSM-MCC. This crystallinity index falls within a range that is typically considered acceptable for pharmaceutical and industrial applications. It suggests a balance between crystalline and amorphous cellulose phases, which is desirable for achieving good mechanical strength in tablets while still allowing for adequate disintegration and dissolution properties. In pharmaceutical formulations, MCC with a crystallinity index of 37% can provide excellent binding and disintegration properties. It helps in forming robust tablets that disintegrate efficiently upon ingestion, releasing the active pharmaceutical ingredient for optimal absorption (Fouad et' al., 2020).

In conclusion, the extracted MSM-MCC sample exhibits micromeritic properties that are generally suitable for pharmaceutical applications, as they align well with standard values typically specified in compendia like the British Pharmacopoeia. These properties ensure the sample's quality, processability, and performance in pharmaceutical formulations (Romruen et al., 2022).

The angle of repose of the extracted MSM-MCC sample was found to be 34°. This indicates that the extracted MSM-MCC powder has moderate to good flow characteristics. Powders with angles of repose in the range of 30° to 35° generally flow well, though they might not be as free-flowing as those with lower angles. Pharmaceutically available microcrystalline Cellulose grades, such as MCC 101 or MCC 102, typically exhibit angles of repose between 20 - 30°. These values indicate excellent flowability and are desirable in tablet and capsule manufacturing for consistent dosing and processing. In effect the

extracted MSM-MCC sample with an angle of repose of 34° has slightly higher flow resistance compared to standard MCC grades. This suggests that while it has good flow properties, it is not as free-flowing as typical pharmaceutical MCC (Romruen et al., 2022).

CHAPTER FIVE

SUMMARY OF FINDINGS, RECOMMENDATIONS AND CONCLUSION

This final chapter summarises the major findings of the study, provides recommendations, and draws conclusions based on the research objectives.

5.1 Summary of Findings, Recommendations and Conclusion

MCC is a multipurpose biopolymer with extensive uses in pharmaceuticals, food, and cosmetics. This project focuses on the extraction and characterization of MSM-MCC, a locally available and underutilized resource. Malted sorghum is rich in cellulose and has significant potential for producing MCC, which can serve as a sustainable alternative to commercially sourced cellulose. The study aims to evaluate the efficiency of the extraction process using acid hydrolysis, assess the physicochemical properties of the obtained MSM-MCC, and explore its potential applications. Through this research, we seek to contribute valuable insights into sustainable practices in MCC production while enhancing the value of malted MSM in industrial applications.

5.2 Summary of Findings

The findings from this study indicate that MCC was successfully extracted from MSM using augmented chemical processes. The extraction and characterization of MCC from MSM yielded a product with a yield of 60%. The particle size of the SEM micrograph of the extracted MSM-MCC was determined using Image J software and found to be 50.5µm.

This makes it a suitable excipient for various industrial applications. The crystallinity index was measured at 86.63%, indicating a high level of crystallinity that can influence its physical properties and reactivity. Thermogravimetric analysis (TGA) demonstrated that the extracted MSM-MCC exhibits good thermal stability within the temperature range of 100 to 275.5 degrees Celsius. This stability suggests that the material can withstand moderate heating without significant degradation. However, a more pronounced thermal degradation was observed between 275.5 and 387.5.

5.3 Recommendations

1. Further studies should be conducted on the MCC extraction process from MSM for further optimisation of the extraction conditions, such as concentration, type of acid/base and duration of treatment, to enhance the yield and purity of the extracted product.
2. A pilot-scale study should be conducted to evaluate the feasibility of MCC commercial production from MSM.
3. Studies should be conducted on the testing of extracted MCC obtained from MSM in various applications, such as in food products or as a pharmaceutical excipient, to assess its performance.

5.3.1 Future Research Direction

The extraction of MCC from other sources of biomass or different varieties of sorghum should be investigated to compare yields and properties.

The chemical modification of the extracted MCC should be explored to enhance its properties for specific applications, such as improving solubility or functionality in formulations. Life cycle assessments are conducted to evaluate the environmental impact of producing MCC from MSM compared to other sources.

A study should be conducted on the potential health benefits or safety implications of using microcrystalline cellulose derived from malted sorghum mash in food or pharmaceutical applications.

Investigate the use of microbial or enzymatic processes for more environmentally friendly extraction and modification of microcrystalline cellulose from malted sorghum mash.

The binding and flow properties of the extracted MSM-MCC need to be assessed. Commercial microcrystalline cellulose is well characterized, whereas the extracted MSM-MCC sample would require rigorous testing to establish its suitability for specific industrial applications.

5.4 Conclusion

Utilizing agricultural waste for the creation of valuable products or raw materials for industrial use is essential in addressing landfill issues. Malted sorghum mash, which is high in cellulose, presents a promising source for industrial applications. Characterization studies have demonstrated the successful extraction of microcrystalline cellulose (MCC) from malted sorghum mash through processes such as pulverization, alkali treatment,

bleaching, and acid hydrolysis. The alkali treatment and hydrogen peroxide bleaching effectively reduced fibre size, leading to an increased surface area of the final product. Additionally, the swelling capacity of the isolated malted sorghum mash MCC (MSM-MCC) can be improved by modifying surface hydroxyl groups, for instance, by adding carboxymethyl groups to produce carboxymethyl cellulose. The MSM-MCC powder produced meets the standards set by the United States Pharmacopoeia (USP) and the British Pharmacopoeia. Consequently, malted sorghum mash serves as a cost-effective source of cellulose, microcrystalline cellulose, and reinforcing materials, suitable for direct use in various industrial and pharmaceutical applications.

REFERENCES

Abdul Khalil, H. P. S., T. Kiat Lai, Y. Y. Tye, M. T. Paridah, M. R. N. Fazita, A. A. Azniwati, R. Dungani, and S. Rizal. (2018). Preparation and characterization of microcrystalline cellulose from sacred Bali bamboo as reinforcing filler in seaweed-based composite film. *Fibers and Polymers* 19 (2), 423–34.

Abu-Thabit, N. Y., A. Abu Judeh, A. S. Hakeem, A. Ul-Hamid, Y. Umar, and A. Ahmad. (2020). Isolation and characterization of microcrystalline cellulose from date seeds (Phoenix Dactylifera L.). *International Journal of Biological Macromolecules* 155(3), 730–39.

Agboeze, E., Theresa O, U., & Ogbobe, O. (2022). Extraction and characterization of pharmaceutical grade microcrystalline cellulose from raphia farinifera inflorescence. *Universal Journal of Pharmaceutical Research*. 7(4), 59-67.

Ansah, I.G.K., Osei-Asare, Y.B., & Donkor, E. (2023). Building resilience of smallholder farmers to climate variability in Ghana. *Hylion,* 9(8), 18556.

Akinoso, R., Ogunjobi, M. A., & Ayoola, A. A. (2022). Microcrystalline cellulose from cassava stalks for pharmaceutical applications. *Journal of Polymers and the Environment,* 30(2), 451–462.

Alebiowu, G., & Itiola, O. A. (2003). The effects of starches on mechanical properties of paracetamol tablets I: Disintegrant. *Acta Pharmaceutica*, 53(3), 231–237.

Asif, M., D. Ahmed, N. Ahmad, M. T. Qamar, N. K. Alruwaili, and S. N. A. Bukhari. (2022). Extraction and characteriza tion of microcrystalline cellulose from Lagenaria siceraria fruit pedicles. *Polymers* 14 (1867), 1–11.

Athirah Abdullah, N., Hanif Sainorudin, M., Asim, N., Mohammad, M., Huda Abd Kadir, N., & Yaakob, Z. (2020). Extraction of Microcrystalline Cellulose from Two Different Agriculture Waste via Chemical Treatment. *Journal of Materials Science and Engineering*, 739(1), 012017

Awika, J. M., & Rooney, L. W. (2004). Sorghum phytochemicals and their potential impact on human health. *Phytochemistry*, 65(9), 1199–1221.

Awika, J. M., Rooney, L. W., Wu, X., Prior, R. L., & Cisneros-Zevallos, L. (2003). Screening methods to measure antioxidant activity of sorghum (Sorghum bicolor) and sorghum products. *Journal of Agricultural and Food Chemistry*, 51(23), 6657–6662.

Azum, N., M. Jawaid, L. Kia Kian, A. Khan, and M. Moteb Alotaibi. (2021). Extraction of microcrystalline cellulose from Washingtonia fibre and its characterization. *Polymers* 13 (18), 1–11.

Belali, N. G., Chaerunisaa, A. Y., & Rusdiana, T. (2019). Isolation and Characterization of Microcrystalline Cellulose Derived from Plants as Excipient in Tablet: A Review. *Indonesian Journal of Pharmaceutics*, 1(2), 23-29.

Beroual, M., Boumaza, L., Mehelli, O., Trache, D., Tarchoun, A. F., & Khimeche, K. (2021). Physicochemical Properties and Thermal Stability of Microcrystalline Cellulose Isolated from Esparto Grass Using Different Delignification Approaches. *Journal of Polymers and the Environment*, 29(1), 130–142.

Dewar, J., Taylor, J. R. N., & Berjak, P. (1997). Determination of improved malting procedures for sorghum. *Journal of Cereal Science*, 26(2), 129–136.

Dicko, M. H., Gruppen, H., Traoré, A. S., Voragen, A. G., & van Berkel, W. J. (2006). Sorghum grain as human food in Africa: relevance of content of starch and amylase activities. *African Journal of Biotechnology*, 5(5), 384–395.

Duodu, K. G., Taylor, J. R. N., Belton, P. S., & Hamaker, B. R. (2003). Factors affecting sorghum protein digestibility. *Journal of Cereal Science*, 38(2), 117–131.

Dykes, L., & Rooney, L. W. (2006). Sorghum and millet phenols and antioxidants. *Journal of Cereal Science*, 44(3), 236–251.

Fathi, H., El-Shazly, A., Elkady, M., Madih, K., (2018). Assessment of New Technique for Production Cellulose Nanocrystals from Agricultural Waste. *Materials Science Forum*, 928 (1662), 83–88.

Fouad, H., L. Kia Kian, M. Jawaid, M. D. Alotaibi, O. Y. Alothman, and M. Hashem. (2020). Characterization of microcrystalline cellulose isolated from conocarpus fiber. *Polymers* 12 (12), 1–11.

Fuseini, I., &Yankson, P. W.K. (2024). Agricultural mechanisation, land tenure and structural transformation in Ghana. Africa Spectrum, 59 (1), 3-25

Gabriel, T., A. Belete, F. Syrowatka, R. H. H. Neubert, and T. Gebre-Mariam. (2020). Extraction and characterization of celluloses from various plant byproducts. *International Journal of Biological Macromolecules*, 158 (264), 1248–58.

George, J., & Sabapathi, S. N. (2015). Cellulose nanocrystals: synthesis, functional properties, and applications. *Nanotechnology, Science and Applications*, 8 (2147), 45–54.

Getachew, M., Gabriel, T., Belete, A., & Gebre-Mariam, T. (2023). Extraction and Characterization of Cellulose and Microcrystalline Cellulose from Teff Straw and Evaluation of the Microcrystalline Cellulose as Tablet Excipient. *Journal of Natural Fibers*, 20(2), 2245565.

Gichuki, J., P. Gachoki Kareru, A. Ngure Gachanja, and C. Ngamau. (2022). Characteristics of microcrystalline cellulose from coir fibers. *Journal of Natural Fibers,* 19 (3), 915–30.

Gnansounou, E., Dauriat, A., & Wyman, C. E. (2005). Refining sweet sorghum to ethanol and sugar: economic trade-offs in the context of North China. *Bioresource Technology,* 96(9), 985–1002.

Hachaichi, A., B. Kouini, L. Kia Kian, M. Asim, and M. Jawaid. (2021). Extraction and characterization of microcrystalline cellulose from date palm fibers using successive chemical treatments. *Journal of Polymers and the Environment,* 29 (6), 1990–99.

Haque, S., Chowdhury, A. A., Rana, A. A., Masum, S., Ferdous, T., Rashid, M. A., Karim, M. M. (2015). Synthesis of microcrystalline cellulose from pretreated cotton obtained from *Bombax ceiba* L. and its characterization. *Bangladesh Journal of Scientific and Industrial Research, 50*(3), 199–204.

Hermin S, Sutriyo, Hasty R S and Dianah R (2017). Preparation of microcrystalline cellulose from water hyacinth powder by enzymatic hydrolysis using cellulose of local isolate *JyoungParm* 99(1), 19-23.

Husin, M., Rahim, N., Ahmad, M. R., Romli, A. Z., & Ilham, Z. (2025). Hydrolysis of microcrystalline cellulose isolated from waste seeds of *Leucaena leucocephala* for glucose production. *Malaysian Journal of Fundamental and Applied Sciences, 15*(2), 1–10.

Hussin, M. H., Pohan, N. A., Garba, Z. N., Kassim, M. J., Rahim, A. A., Brosse, N., & Haafiz, M. K. M. (2016). Physicochemical properties of microcrystalline cellulose from oil palm fronds as potential methylene blue adsorbents. *International Journal of Biological Macromolecules,* 92 (141), 11–19.

Kalita, R. D., Nath, Y., Ochubiojo, M. E., & Buragohain, A. K. (2013). Extraction and characterization of microcrystalline cellulose from fodder grass (*Setaria glauca* (L.) P. Beauv) and its potential as a drug delivery vehicle for isoniazid. *Colloids and Surfaces B: Biointerfaces,* 108 (1016), 85–89.

Karna, A., Thakur, S., & Shrestha, B. (2022). Extraction and characterization of cellulose from agricultural residues: Wheat straw and sugarcane bagasse. *Journal of Nepal Chemical Society*, 43(1), 93–98.

Katakojwala, R.; Mohan, S.V. (2020). Microcrystalline cellulose production from sugarcane bagasse: Sustainable process development and life cycle assessment. *J. Clean. Prod*. 249 (959), 119342.

Kharismi, R. R. A. Y., Sutriyo, & Suryadi, H. (2018). Preparation and characterization of microcrystalline cellulose produced from Betung bamboo (*Dendrocalamus asper*) through acid hydrolysis. *Journal of Young Pharmacists, 10*(2), 79–83.

Kian, L. K., Jawaid, M., Ariffin, H., & Alothman, O. Y. (2017). Isolation and characterization of microcrystalline cellulose from roselle fibers. *International Journal of Biological Macromolecules,* 103 (141), 931–940.

Kian, L. K., Saba, N., Jawaid, M., & Fouad, H. (2020). Characterization of microcrystalline cellulose extracted from olive fiber. *International Journal of Biological Macromolecules,* 156 (8130), 347–353.

Klemm, D., Kramer, F., Moritz, S., Lindström, T., Ankerfors, M., Gray, D., & Dorris, A. (2011). Nanocelluloses: a new family of nature-based materials. *Angewandte Chemie International Edition,* 50(24), 5438–5466.

Kunusa, W. R., Isa, I., Laliyo, L. A. R., & Iyabu, H. (2018). FTIR, XRD and SEM Analysis of Microcrystalline Cellulose (MCC) Fibers from Corncorbs in Alkaline Treatment. *Journal of Physics: Conference Series,* 1028(1),012199.

Lai, S. M., Huang, C. K., & Shen, H. F. (2005). Preparation and properties of biodegradable poly (butylene succinate)/starch blends. *Journal of Applied Polymer Science,* 97(1), 257–264.

Liu, Y., A. Liu, S. A. Ibrahim, H. Yang, and W. Huang. (2018). Isolation and characterization of microcrystalline cellulose from pomelo peel. International Journal of Biological Macromolecules 111(8130), 717–721.

Liu, Z., Wang, Z., Yang, S., Ji, N., & Li, D. (2025). Extraction process research and characterization of microcrystalline cellulose derived from bamboo (*Phyllostachys edulis* fibers). *Polymers,* 17 (9), 1143.

Misgana, T., B. U. Chaudhary, and R. D. Kale. (2019). Extraction and analysis of microcrystalline cellulose from delignified serte leaf fiber wastes. *Journal of Natural Fibers* 18 (11):1729–1741.

Mohamad Haafiz, M. K., S. J. Eichhorn, A. Hassan, and M. Jawaid. (2013). Isolation and characterization of microcrystalline cellulose from oil palm biomass residue. *Carbohydrate Polymers* 93 (2), 628–634.

Mohamed El-Sakhawy & Mohammad L. Hassan (2007)**.** Physical and mechanical properties of microcrystalline cellulose prepared from agricultural residues. *Carbohydrate Polymers*, 67 (1), 1-10.

Naduparambath, S., and E. Purushothaman. (2016). Sago seed shell: Determination of the composition and isolation of microcrystalline cellulose (MCC). *Cellulose*, 23 (3), 1803–1812.

Nazir, S., Habib, U., & Ul Islam, T. (2023). Extraction and Characterization of Microcrystalline Cellulose from Walnut, Almond and Apricot Stone Shells. *Journal of the Chemical Society of Pakistan*, 45(3), 85–92.

Ohwoavworhua, F. O., & Adelakun, T. A. (2010). Non-wood fibre production of microcrystalline cellulose from *Sorghum caudatum*: Characterisation and tableting properties. *Indian Journal of Pharmaceutical Sciences,* 72(3), 295–301.

Okoye, E. I., Onunkwo, G. C., Kunle, O. O., & Okoli, P. C. (2015). Comparative evaluation of the flow and compaction characteristics of α-cellulose and microcrystalline cellulose powders derived from groundnut husk. *International Journal of Pharmaceutical Sciences Review and Research*, 30(1), 116–120.

Olugbenga, O., Labunmi, L., & Bodunde, O. (2014). Microcrystalline cellulose from plant wastes through sodium hydroxide-anthraquinone-ethanol pulping. *BioResources,* 9(4), 6166–6192.

Qinfeng, H., Q. Wang, H. Zhou, D. Ren, H. Yunhai, H. Cong, and W. Long. (2018). Highly crystalline cellulose from brown seaweed saccharina japonica: Isolation, characterization and microcrystallization. *Cellulose* 25 (10), 5523–5533.

Rasheed, M., Jawaid, M., Karim, Z., & Abdullah, L. C. (2020). Morphological, physiochemical and thermal properties of microcrystalline cellulose (MCC) extracted from bamboo fiber. *Molecules,* 25(12), 2824.

Reddy, B. V. S., Ramesh, S., Reddy, P. S., & Ramaiah, B. (2005). Sweet sorghum: A potential alternate raw material for bio-ethanol and bio-energy. *International Sorghum and Millets Newsletter*, 46 (2), 79–86.

Rojas, J., & Kumar, V. (2012). Comparative evaluation of microcrystalline cellulose II powders. *International Journal of Pharmaceutics*, 422(2), 160–165.

Romruen, O.; Karbowiak, T.; Tongdeesoontorn, W.; Shiekh, K.A.; Rawdkuen, S. (2022). Extraction and Characterization of Cellulose from Agricultural By-Products of Chiang Rai Province, Thailand. *Polymers*. 14(9), 1830.

Saeed, F., Afzaal, M., Tufail, T., & Sultan, M. T. (2020). Dietary fiber and cardiovascular health: A review. *International Journal of Food Properties*, 23(1), 1577–1592.

Saini, J. K., Patel, A. K., Adsul, M., & Singhania, R. R. (2016). Cellulase adsorption on lignin: A roadblock for economic hydrolysis of biomass. *Renewable Energy*, 98 (960), 29–42.

Sainorudin, M. H., Mohammad, M., & Kadir, N. H. A. (2018). Characterization of several microcrystalline cellulose (MCC)-based agricultural wastes via X-ray diffraction method. *Solid State Phenomena*, 280 (1662), 340–345.

Saulnier, L., Sado, P. E., Branlard, G., Charmet, G., & Guillon, F. (2007). Wheat arabinoxylans: Exploiting variation in amount and composition to develop enhanced varieties. *Journal of Cereal Science*, 46(3), 261–281

Senusi, N. A., Shohaimi, N. A. M., Halim, A. Z. A., Shukr, N. M., Azhar Abdul Razab, M. K., Mohamed, M., Amin, M. A. M., Wan Mokhtar, W. N. A., Ismardi, A., & Abdullah, N. H. (2020). Preparation & characterization of microcrystalline cellulose from agriculture waste. *IOP Conference Series: Earth and Environmental Science*, 596(1), 012035.

Shi, S., Zhang, M., Ling, C., Hou, W., and Yan, Z. (2018). Extraction and characterization of microcrystalline cellulose from Waste Cotton fabrics via hydrothermal method. *Waste Management* 8, 139–146.

Singh Pal, G., Madiwale, P. V., & Adivarekar, R. V. (2017). Preparation and characterization of microcrystalline cellulose (MCC) from renewable source. *Current Applied Polymer Science,* 1(2), 1–10.

Siro, I., & Plackett, D. (2010). Microfibrillated cellulose and new nanocomposite materials*: a review*. *Cellulose*, 17(3), 459–494.

Sonia, A., & Dasan, K. P. (2013). Chemical, morphology and thermal evaluation of cellulose microfibers obtained from *Hibiscus sabdariffa*. *Carbohydrate Polymers,* 92(3), 668–674.

Tan, W. Y., Gopinath, S. C., Anbu, P., Velusamy, P., Gunny, N., Anas, A., & Subramaniam, S. (2023). Generation of microcrystalline cellulose from cotton waste and its properties. *BioResources*, 18(3), 4884–4896.

Tarchoun, A. F., D. Trache, and T. M. Klapötke. (2019). Microcrystalline cellulose from posidonia oceanica brown algae: Extraction and characterization. *International Journal of Biological Macromolecules* 138, 837–45.

Taylor, J. R. N., Schober, T. J., & Bean, S. R. (2006). Novel food and non-food use for sorghum and millets. *Journal of Cereal Science*, 44(3), 252–271.

Tessema, T. A., Feroche, A. T., Workneh, G. A., & Gabriel, T. (2023). Physicochemical Characterization of Cellulose and Microcrystalline Cellulose from Cordia africana Lam. Seeds. *Journal of Natural Fibers*, 20(2), 2198278.

Trache, D., Hussin, M. H., Chuin, C. T. H., Sabar, S., Fazita, M. R. N., Taiwo, O. F. A., Haafiz, M. K. M. (2016). Microcrystalline cellulose: Isolation, characterization and bio-composites application—A review. *International Journal of Biological Macromolecules*, 93, 789–804.

Trache, D., Hussin, M. H., Haafiz, M. K. M., & Thakur, V. K. (2017). Recent progress in cellulose nanocrystals: sources and production. *Nanoscale*, 9(5), 1763–1786.

Uesu, N. Y., Pineda, E. A. G., & Hechenleitner, A. A. W. (2000). Microcrystalline cellulose from soybean husk: Effects of solvent treatments on its properties as acetylsalicylic acid carrier. *International Journal of Pharmaceutics*, 206, 85–96.

Ul-Islam, M., Khan, T., & Park, J. K. (2012). Nanoreinforced bacterial cellulose-montmorillonite composites for biomedical applications. *Carbohydrate Polymers*, 89(4), 1189–1197.

Uzondu, A. W., Okafor, I. S., & Uzochukwu, A. I. (2022). Evaluation of physicochemical and flow properties of microcrystalline cellulose derived from maize stalk. *Nigerian Journal of Pharmaceutical and Applied Science Research*, 11(1), 45–52.

Veeramachineni, A. K., T. Sathasivam, S. Muniyandy, P. Janarthanan, S. James Langford, and L. Yau Yan. (2016). Optimizing extraction of cellulose and synthesizing pharmaceutical grade carboxymethyl sago cellulose from Malaysian sago pulp. *Applied Sciences (Switzerland)* 6 (6):170–88.

Zegada-Lizarazu, W., & Monti, A. (2012). Are we ready to cultivate sweet sorghum as a bioenergy feedstock? A review on field management practices. *Biomass and Bioenergy*, 40 (961), 1–12.

Zeleke, N. M., Sinha, D. K., & Mengesha, G. A. (2022). Chemical Composition and Extraction of Micro Crystalline Cellulose from Outer Skin Isolated Coffee Husk. *Advances in Materials Science and Engineering*, 2022, 1-13.

Zeni, M., & Favero, D. (2016). Preparation of microcellulose (MCC) and nanocellulose (NCC) from Eucalyptus kraft pulp. *Polymer Science,* 1, 1–5..

Zhang, Y., X. Yongjian, X. Yue, L. Dai, and N. Yonghao. (2019). Isolation and characterization of microcrystalline cellulose from bamboo pulp through extremely low acid hydrolysis. *Journal of Wood Chemistry and Technology*, 39 (4), 242–54.

Zhao, T., Chen, Z., Lin, X., Ren, Z., Li, B., & Zhang, Y. (2018). Preparation and characterization of microcrystalline cellulose (MCC) from tea waste. *Carbohydrate Polymers,* 184, 164–170.