Manufacturing and Characterization of Millet Starch-Based Superabsorbent Polymer

Thesis Submitted to

The Superior University Lahore

In Partial Fulfillment of the

Requirement for the Degree of

Master of Philosophy in Chemistry

By

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Roll No. MSCHE-F21-006

Session: 2021-2023

Faculty of Sciences

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Dedication

In the name of Allah, the Most Gracious, the Most Merciful. To the Beloved Messenger of Allah, the Seal of Prophets, the Mercy to the Worlds, the embodiment of compassion and wisdom, I humbly dedicate this thesis research, a humble endeavor in the realm of knowledge, to your blessed memory and unparalleled legacy. O Prophet of Allah, you were the epitome of love, justice, and mercy. Your noble character, profound teachings, and exemplary life continue to illuminate the path of humanity, guiding us towards righteousness and enlightenment. Through your divine guidance, you brought forth a message of unity, peace, and social justice. You championed the rights of the oppressed, uplifted the downtrodden, and fostered a society built on compassion and equality. Your wisdom transcends time and encompasses every aspect of life, from matters of faith and spirituality to governance, ethics, and human interactions. You taught us the importance of seeking knowledge, expanding our horizons, and embracing intellectual pursuits as a means of attaining closeness to our Creator. You demonstrated the true essence of leadership, humility, and selflessness, and set an unparalleled example of how to navigate the complexities of life with grace and unwavering faith. As I embark on this academic journey, I seek to honor your blessed memory by striving for excellence, integrity, and a deep commitment to truth. I pray that my research may contribute, in even the slightest way, to the betterment of society, guided by the timeless principles you imparted. I beseech Allah, the Most Merciful, to accept this humble dedication and make it a source of blessings and enlightenment. May He grant me the strength and wisdom to tread the path of knowledge with sincerity and humility, following in the footsteps of the greatest teacher and guide that humanity has ever known. Indeed, in your blessed existence, we find solace, guidance, and inspiration. Your life is a testament to the limitless potential of the human spirit, and your teachings continue to resonate across generations, transcending barriers of time, culture, and geography. May peace and blessings be upon you, O Prophet of Allah, forever and always.

Acknowledgement

All praise and gratitude are due to Allah, the Lord of all creation, the Most Merciful, and the Bestower of knowledge and wisdom. I begin by acknowledging His infinite blessings and expressing my deepest gratitude for guiding me throughout this remarkable journey of research.

I extend my heartfelt appreciation and thanks to the Holy Prophet Muhammad (peace be upon him), the ultimate source of inspiration and guidance. His profound teachings and noble character have been the beacon of light that illuminated my path, enabling me to navigate the complexities of this research endeavor.

To my parents, your endless love, sacrifices, and belief in my abilities have been the foundation of my success. Your constant encouragement and understanding during challenging times have been invaluable, and I am forever grateful for your unwavering support.

I would like to express my sincere gratitude to my research supervisor, Dr. Hina Zain, for her invaluable guidance, unwavering support, and scholarly expertise throughout this remarkable term of research. Her profound insights, intellectual acumen, and meticulous attention to detail have greatly enriched the quality and depth of this thesis.

Worthy Program Leader, Dr. Muhammad Mudassir Iqbal did great effort in offering a set of attributes for our research and enforced rules and regulations which resulted in great fruit which will benefit me in the future. In addition to it, I also offer thanks and gratitude to Dean of Faculty of Sciences, Prof. Dr. M. Naveed Babur and Head of the Department of Biological Sciences, Prof. Dr. Uqba Mehmood, for providing and maintaining quality education and a conducive environment for research.

I humbly submit this thesis as a testament to the mercy and guidance of Allah and as a humble expression of gratitude to the Holy Prophet Muhammad (peace be upon him) and my esteemed supervisor, Dr. Hina Zain.

Asad Ali Tariq

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List of Abbreviations

Superabsorbent Polymer	SAP
Pearl Millet Starch-based Superabsorbent Polymer	PMSSAP
Acrylic Acid	AA
Acrylamide	AM
Potassium Persulfate	KPS
Sodium Hydroxide	NaOH
N, N'-Methylene-Bisacrylamide	MBA
Potassium Hydroxide	KOH
Carboxymethyl Cellulose	CMC
Fourier Transform Infrared Spectroscopy	FTIR
Scanning Electron Microscopy	SEM
Thermogravimetric Analysis	TGA
X-Ray Diffraction	XRD
Transmission Electron Microscopy	TEM
Differential Scanning Calorimetry	DSC
Gel Layer Permeability	GLP
absorbance Under Load	AUL
Mimosa Pudica Hydrogel	MPH
Field Emission Scanning Electron Microscopy	FE-SEM
Electrospun Cellulose Nanofibers	ECNFs
Hydroxyethyl cellulose	HEC
Nitrocellulose	NC
Centrifuge Retention Capacity	CRC
Dopamine	DA
Sugarcane Bagasse Cellulose	SCB

ABSTRACT

Pearl Millet Starch-based Superabsorbent Polymer (PMSSAP) with good absorption properties was synthesized by using monomers acrylic acid (AA) and acrylamide (AM), cross-linker methylene bis-acrylamide (MBA), and activator tetramethyl ethylene diamine, along with the initiator potassium persulfate (KPS). Millet seeds were soaked in 0.25% NaOH for 24 hours and then pulverized methods was employed which resulted a fine millet starch, that is the significant ingredient of desired polymer. FTIR was used to confirm the functional group and TGA technique was used to check thermal stability of superabsorbent polymer. SEM and XRD were used to examine the morphology and crystal structure of PMSSAP, respectively. The pH of PMSSAP is 8.3 at ambient temperature. Physiological-chemical tests were conducted to evaluate its performance, such 0.05 % of MBA shows highest absorbance at 345 g/g. The PMSSAP showed considerable water absorption when submerged in pure water, which contains no salt. However, even trace levels of salt in the system dramatically reduced its capacity to absorb water. Superabsorbent polymers are impacted by temperature because it changes their ability to swell. Lower temperatures improve absorption, but higher temperatures can break hydrogen bonds and decrease water retention. The cross-linking density and polymer composition affect thermal stability. Many superabsorbent polymer, especially polymer-based absorbent materials, have been effectively produced in recent years, exhibiting remarkable water absorption and retention capacities. Materials for water absorption and retention are highly sought after in a variety of industries, including as agriculture, controlled medicine release systems, personal hygiene products, and baby diapers. Water absorbance capacity is a crucial metric for assessing polymer-based absorbents, which have found use in several fields as a novel moisture-absorbing substance. Grams of liquid absorbed per gram of dry polymer was measure of the water absorbance capacity in this investigation. In conclusion, the preparation of biodegradable superabsorbent polymers presents a promising avenue for sustainable applications, combining effective water absorption with environmentally friendly characteristics.

CHAPTER 1

INTRODUCTION

The polymer has several molecules, which include various chemical units. These repeating chemical units are called monomers. According to their source, natural polymers, semisynthetic polymers, and synthetic polymers are the three main groups of polymers. Animals, plants, and microscopic creatures all contribute to the creation of natural polymers. Carbohydrates and proteins are natural polymers that help sustain the structural integrity of plants and animals. The six primary categories of natural polymers are lignin, proteins, polysaccharides, polynucleotides, polyisoprenes, and polyesters. Natural polymers have several advantages over the other two polymer types, including cost benefits, accessibility, the possibility of biodegradability, and biocompatibility as a result of their natural origin. (1)

Super absorbent polymers (SAPs) are getting sought-after these days because of their high-water retention and being environmentally friendly. SAP can systematize by cross-linking and lining up into three-dimensional patterns. Through hydrogen bonding, SAP is capable of absorbing aqueous fluid up to 100 times its weight. The absorbed liquid then accumulates in the pores of the molecular polymeric network. Eventually, gel-like material is produced to seal off the absorbed fluid. (2)

Conventional hydrogels with a maximum water absorption capacity of 1 gram per gram are called superabsorbent polymers, differentiated by their remarkable capacity to absorb water at levels ranging from 10 to 1000 grams per gram of deionized water. Depending on the application for which they are designed, SAPs must satisfy several requirements to be considered ideal. These requirements include the ability to regulate both adsorption and absorption rates, exceptional water retention even under load, a high degree of gel formation following cross-linking, cost-effectiveness, enduring tenacity and steadfastness during swelling or storage, odor absence, maximum biodegradability, being non-toxic, and being able to gradually release absorbed liquids. Adsorption and absorption are the two basic processes that may be used to explain the mechanism underlying SAP swelling. The solvent or water molecules that are absorbed into the polymeric network structure of

SAPs cause expansion as a result of the interconnected polymer chains. This new molecular arrangement therefore produces a flexible and contracting force. (3)

The market is now dominated by two main kinds of SAPs: those made from synthetic petrochemical sources and those made from natural polysaccharides and polypeptides. Because of its superior cost-effectiveness ratio, cross-linked and partly neutralized sodium polyacrylate is the most typical and commonly used super absorbent polymer form in commercial applications. However, it's important to remember that this specific SAP type lacks biodegradability and has a remarkably large molecular weight. (4) These are called non-biodegradable SAPs, possessing high absorption capacity with different compositions of acrylic acid (AA) and their salts, acrylamide (AM) and acrylonitrile (AN). Radical chain growth polymerization is carried out in the formation of nonbiodegradable carbon-to-carbon bond SAPs which consist of the cross-linking and the main chain of the polymer. Biodegradation follows disintegration and breakdown of a body or thing with the assistance of microorganisms that yield new biomass, the mineralization of organic compounds, and the reusing of CO_2 gas. Water, carbon dioxide, methane gas, and nitrogen gas evolved during biodegradation.

Plastic garbage that can be recycled is only 9% of total plastic garbage. Whereas only 10% of plastic garbage is combusted, 80% of plastic garbage is discarded in open places. Methane gas evolved during the breakdown of disposable pamper which increased the temperature of the environment by displacing oxygen gas. This phenomenon leads to global warming. It has been noticed that SAPs also contaminate the air quality, during the production, delivery, and disposal of pampers. Control the chemicals that contaminate the air quality such as sodium polyacrylate, tributyltin, chlorine, and polychlorinated-pdioxins.

A massive amount of wastewater is released into the manholes during the preparation of SAPs, the water has polychlorinated-p-dioxins, polychlorinated dibenzofuran, heavy metals, sludge, solvents, and chlorophenols like toxic chemicals that pollute the underground water. Disposable pampers are being thrown anywhere for a long span without noticing that it is the reason for land pollution. Plastics and SAPs gel require centuries to break down in the presence of the sun and air.

The duration of the interaction of the pampers with the baby's skin is normally around 24 hours in a whole day. That causes serious health issues for children. Children's skin does not have immunity to tackle dermatological problems such as rashes, acne, and hives because of synthetic chemicals such as xenobiotic chemicals. Xenobiotic chemicals used in preparing SAP are unsuitable for children's skin.

Currently, biodegradable SAPs are being developed by keeping all the above-mentioned defects and problems caused by synthetic SAPs. Starch, being a natural substance, gets biodegraded, whereas acrylates, being synthetic, become non-biodegraded, which is 50% of SAP's total formulations. (5)

A notable polysaccharide that is abundantly present in plants that are naturally occurring, renewable, biocompatible, and non-toxic is starch, which is predominantly derived from millet, cassava, wheat, rice, corn, and potatoes. The many hydroxyl groups in the starch backbone have strong attraction for other hydroxyl groups, which makes it easier for water molecules to establish hydrogen bonds with them. Starch chains interact with water molecules during the gelatinization process, causing the chains to split and the starch granule to enlarge. Raw starch is frequently mixed with more hydrophilic polymers to increase liquid absorption capacity and counteract the starch's natural hydrophobicity due to its granular nature.

To increase its adaptability in a variety of applications, starch is subjected to chemical, physical, or biological changes. The manufacture of starch-based SAP has received significant scientific attention, frequently in conjunction with synthetic polymers and biopolymers, including proteins and polysaccharides. The characteristics and utility of starch-based SAP are greatly enhanced by the addition of several polymer classes. (3)

Amylopectin has a linear polymer of α-(1-4) glucose units with periodic branches of α-(1-6) links, in contrast to amylose's linear structure of α- (1-4) glucose units. Starch and its derivatives' intrinsic qualities have been employed in a variety of industrial sectors, like cosmetics, food, and pharmacy. (6)

Pearl millet starch contains fat, protein, carbohydrate, moisture, ash, and fiber with a chemical composition of 2.2±0.21 %, 12.8±0.30%, 71.9±0.50%, 9.59±0.25%, 1.53±0.12%, and 2.0±011%.0.33 respectively. Millions of tons of pearl millet are

cultivated, making it the 6th-ranking cereal in Pakistan. Pearl millet is cultivated in various cities in Punjab, notably in Dera Ghazi Khan and Gujranwala. (7)

In Japan, superabsorbent polymers were initially made available for purchase in 1978, and their primary utilization was in sanitary pads. A polyacrylate that had been starch grafted and cross-linked was the first superabsorbent substance. The older super absorbents were gradually supplanted by polyacrylic acids throughout time, and these days, SAPs are primarily made of polyacrylic acids. European nations were instrumental in the advancement of SAP technology for use in infant diapers in 1980. The initial diapers that made use of this technique had only a modest amount of polymer—roughly one to two grams. In Japan, a more refined diaper with 4–5 grams of polymer and less fluff was released onto the market by 1983. (8)

The potential of pearl millet starches as a suitable replacement for traditional starch sources like corn, rice, and potato, which are frequently used to make food, has to be further investigated. A new direction for the starch business is the creation of biodegradable SAP utilizing pearl millet starches. Pearl millet starch has a rather limited range of uses for both culinary and non-food applications. As a result, the current study started to look at the unique qualities of pearl millet starch and assess their feasibility for making Pearl Millet Starch-based Superabsorbent Polymer (PMSSAP).

The grain pearl millet is the most adaptable grain, being inexpensive, widely available, high in starch, remarkably durable, low price everywhere throughout the world. This work then thoroughly analyzed production and characterization of a new superabsorbent polymer. From methodical design and optimization of the synthesis process, an outstandingly water absorbing polymer and very efficient system emerges. With a combination of many analytical techniques, such as Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), thermogravimetric analysis (TGA), X-ray diffraction (XRD), the structure and morphology of the superabsorbent polymer are clarified. But the outcomes also immediately validate the fact that the polymer was successfully produced, and provide valuable new details about it that could not otherwise have been known. The results provided contribute to building up the knowledge corpus in polymer science and have applied potential for various domains.

The synthesis and characterization techniques yield the evidence of effective synthesis and characterization techniques for an enhanced functionality and precisely defined properties of produced superabsorbent polymer, which will open the door to other developments in production of the high performing water absorbing materials, consumer goods, wastewater treatment and agriculture.

Present study

Quality of the pearl millet starch-based super absorbant polymer (PMSSAP) was examined using several methods such as Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), thermogravimetirc (TGA) and X-ray diffraction (XRD). However, the product's swell ability as well as ability of absorption were also part of the quality assessment. To make 45.5g of the starch powder, 70g of pearl millet is soaked in 0.25% NaOH solution. This extracted starch was further used to create the superabsorbent polymer using pearl millet starch. Since Pearl Millet Starch based Superabsorbent Polymer (PMSSAP) is available and affordable, it was intentional to choose for synthesis of PMSSAP. The thorough examination of applicability and prospective applications with the use of cutting-edge analytical techniques and performance testing shows feasibility for many practical tasks.

AIMS AND OBJECTIVES

I. Synthesis of superabsorbent polymer by pearl millet starch.

II. Evaluate its structural, thermal, and absorbance properties through advanced characterization techniques.

CHAPTER 2

LITERATURE REVIEW

Using 10 MeV electron beam irradiation, Zhang Sufen et al. (2014) actually made and characterized a starch-grafted superabsorbent. Alkaline hydrolysate and concurrent electron beam irradiation of ambient temperature (10 MeV) produced the superabsorbent polymer starch-graft-polyacrylamide cross linked with N, N'-methyl bisacrylamide mixture (MBA). The effect of the irradiation dosage, acrylamide to anhydroglucose unit ratio and the cross-linker concentration on the properties of resulting polymers was investigated systematically. Optimization testing indicated that the optimal parameters were an 8 kGy total dosage, a 4.5 mol/mol acrylamide-to-anhydroglucose unit ratio, and a 0.4% mol/mol crosslinker-to-acrylamide ratio, the superabsorbent polymer exhibited high absorption capacity of 1452 g/g for pure water and 83 g/g calculated on dry weight basis for a saline solution. The results show that 10 MeV electron beam irradiation rather than gamma ray irradiation is more effective due to its higher energy as well as with dosage rate. (9)

Akhmad Zainal Abidin et al. (2014) has done a superabsorbent polymer composite which copolymerised using cassava starch. A bentonite acrylate acid composite superabsorbent polymer (SABC) which was fabricated by copolymerizing cassava starch as its major component was developed. SAPC was prepared by graft polymerization and bentonite at nanoscale as reinforcing agent. The experimental considerations for this study were the amount of the acrylic acids to starch weight ratio, the amount of the weight percent of the initiator, and the amount of cross link range. FTIR, SEM and TGA DSC techniques were used to determine the characteristics of the product. This study demonstrated that the polymerization processes took place in two crucial phases: the incorporation of acrylic acid monomers to the starch chains as the structural foundation of the polymer. Of notable importance though, was that the addition of cassava starch significantly increased the speed with which the polymerization procedure was performed: average reaction times were from 10 to 15 minutes. More importantly, this efficacy enabled the more cost effective manufacture of SAPC due to this. Additionally, the addition of bentonite was necessary for the improvement of thermal characteristics of the finished products by

interacting with monomers by hydrogen bonds and weak bonds. The acrylic acid to starch weight ratio was changed to 5, initiator was 0.5%, cross linker was 0.05%, and bentonite concentration was 2%. Because the product actually is versatile, a range of sectors, including medicine, farming, traditional superabsorbent polymer uses, may seek to use the final product. (10)

Mengmeng Diao et al. (2014) worked on the formation and adsorption characteristics of peanut hull composite and superabsorbent hydrogel. Using a solution polymerization process, a novel composite material made of a superabsorbent hydrogel and peanut husk was created. Through orthogonal testing, it was discovered what the ideal circumstances for adsorbing Pb(II) were: a neutralization level of 70%, a cross-linker dosage of 0.6%, an initiator dosage of 0.4 %, and a peanut husk dosage of 10% about the monomers. In saline solution, deionized water, and tap water, he observed how this composite material expanded. Pb(II) from aqueous solutions was further eliminated using the composite. Adsorption equilibrium could be achieved in 60 minutes, according to kinetic measurements, and the procedure followed a pseudo-second-order model. The adsorption isotherms were elucidated by the Freundlich isothermal model. The adsorption process was both crucially endothermic and spontaneous (11)

Carboxymethylcellulose (CMC) and hydroxyethylcellulose (HEC) were used to develop hydrogels as reported by Yoldas Seki et al. (2014). A novel hydrogel was developed with a pH and salinity sensitivity using CMC and HEC. This synthesis was performed by using malic acid and fumaric acid in different amounts. Variations of pH, adding salt to the water, or adding other fluids, all of these were tested on the capacity of these hydrogels to soak up water. In the pH dependent experiments, the hydrogels were seen to have higher water absorption capacities when the hydrogels have been increased (pH 7.4), suggesting reversible pH sensitivity in CMC-HEC based hydrogels. Furthermore, the water absorption capacity of these hydrogels varied in accordance with the increase in the charge of the metal cation (Al^{3+}, Ca^{2+}, or Na^{+}) showed that the hydrogels are sensitive to the metal ions. Tensile tests data of hydrogels under tensile condition shows that higher tensile strength values were achieved by increasing the concentrations of cross-linker. Additionally, scanning electron microscopy pictures were used to investigate

morphological properties and thermogravimetric analysis to estimate samples thermal stability. (12)

Agricultural applications of superabsorbent polymers were studied by Enas M. Ahmed et al. (2015) with synthesis and characterization of the polymers. In this investigation, the effect of changes in potassium persulfate used as an initiator, cross linker (MBAM), and AA/KOH ratio on the superabsorbent hydrogel swelling characteristics was investigated. The following parameters were found to be optimal for the production of an economical and efficient superabsorbent hydrogel: AA = 60.2%; KOH = 37.4%; KPS = 2%; MBAM =0.4%; and an AA/KOH ratio of 0.9 mole/mol. With a yield of roughly 92.2%, superabsorbent hydrogels were produced on a bench scale under these ideal circumstances. (13)

According to Hamid-Reza Fallahi et al. (2016), superabsorbent polymers, the size of the mother corm with respect to the saffron's output, stigma output, flower yield, and replacement corms is a vital factor to be taken note of in the production of saffron. However, growth of the best saffron corms is limited by water scarcity in semi-arid area. In this study the effect of superabsorbent polymer on replacement corm production was studied where there were 6 different levels applied at 0 to 50 kg/ha..According to the initial findings, corm growth indices were unaffected by SAP use during the first life cycle of saffron. But as the life cycle went on, SAP's advantages became more obvious. Specifically, the application of 40 kg/ha SAP increased the scale weight of corms per clone by 50%, the quantity of replacement corms per clone by 13%, and the average weight of replacement corms per clone by 36% as compared to the control. Additionally, the mean weight of replacement corms and the mean number of buds per corm were 29% and 27% higher, respectively, in the 40 kg ha-1 handling of superabsorbent polymers compared to the control. The SAP application reduced the output of non-standard corms (less than 8 g) and significantly increased the percentage of corms with standard weights. Overall, the quantity of corms produced in each clone was less positively impacted by SAP than corm weight, bud count, and standard corm yield. Furthermore, the water-use efficiency of the SAP application treatments (6.1 kg standard corms per cubic meter) was greater than that of the control treatment (4.34 kg per cubic meter). (14)

Adina Matica et al. (2017) have investigated the biodegradability of chitosan products. In both in vitro and vivo chitosan biodegradation requires either enzymatic or chemical catalysis of the polymer. However, in this process, we break down the polymer substance into simpler parts, that is monomers like D-glucosamine and N-acetyl-glucosamine. Biodegradation rate of chitosan is controlled by the amounts of deacetylation (DD) and molecular weight (MW). The principal functions of breaking the chitosan and derivatives are catalyzed by lysozymes, chitosanase, chitinase, and chitin deacetylase which hydrolyze the chitosan and cause those biomaterials to biodegrade in the human body. Degree of deacetylation and molecular weight of the chitosan is the main variables controlling the rate of biodegradation of the chitosan. These factors are generally accepted to be rapid the lower and slow the higher values, and to speed up biodegradation and slow it down. (15)

Eggshell, acrylic copolymer and the addition of biofiller were determined to be used or preparing a composite superabsorbent hydrogel by Marcos Vinícius A. Queirós et al. (2017). Eggshell (ES) hydrogel composite, a superabsorbent hydrogel was developed using waste material which is mainly calcium carbonate and available anywhere. Comparisons of this composite to the same gel but without filler, and used a poly (acrylamide-co-potassium acrylate) matrix (17 weight % filler, chicken ES filler), were made. It was discovered that the dispersal of the ES within the polymeric matrix was uniform, and FTIR examination showed that the acrylate and calcium cations interacted. With a noteworthy 100% increase in water absorption and a 41% increase in saline solution absorption, this composite showed improved gel strength. This composite is ideal for agricultural applications because of its favorable qualities, which include extensive swelling, a homogeneous structure, and strong mechanical capabilities brought on by the inclusion of a sizable amount of cost-effective waste material. Additionally, this strategy provides a workable and environmentally responsible way to reuse eggshell waste. (16)
Amjad Ali et al. (2017) created and described composite starch-based films strengthened with hulls from corn and wheat. A solution-casting technique was used to create films made with starch that were strengthened by two types of natural materials along with secure reinforcing agents obtained from wheat as well as maize hulls. The inclusion of these hulls improved the starch matrix's modulus, bending strength, and damage

toughness. The significant improvement in impact strength can be primarily ascribed to the higher load force, demonstrating that the inclusion of stiff fillers strengthened the materials as expected. Additionally, the addition of hulls improved the starch films' ability to act as a gas barrier, especially maize hulls, which have higher density and more crystallinity due to their flat structural makeup. When compared to fillers with bigger particles, films that are strengthened with fillers that have smaller particles showed higher mechanical performance. Starch and both kinds of hulls have a high level of compatibility, as studied using scanning electron microscopy. Following processing, the two types of hulls' crystal arrangements in the films stayed unchanged, which was shown by regular and polarized optical microscopy investigations, illuminating the reinforcement mechanism. Additionally, the capacity of the films made using starch was enhanced by the inclusion of grain byproducts to block UV light. (17)

Nur Raihan Mohamed et al. (2018) proposed a method using derivatives of polyvinyl alcohol and starch derived from mung bean SAP in the presence of coupling agents like maleic anhydride. The swelling percentage is dependent on the quantity of starch. Maleated polyvinyl acetate: mung bean mixture with a 70:30 ratio exhibits the highest initial water absorption % in a water absorption test, whereas a 50:50 ratio produces the lowest results. Due to starch loss from the hydrogel, the addition of starch to maleated polyvinyl acetate lowers water absorption. In contrast to hydrogels with a 50:50 to maleated polyvinyl acetate: mung bean ratio, those without additional starch (100:0) had the lowest swelling capacities. Whereas a high quantity of starch favors the biodegradation process. The greatest capability of swelling for the hydrogel occurred at a 70:30 ratio, swelling 491%. It was found that 20% and 60% are the maximum amount of starch to obtain maximal swelling percentage. In conclusion, the hydrogel could be used in water absorbing applications due to large capacity of swelling in the 70:30 ratios. (18)

According to their synthesis and characterization, Jungmin Lee et al. (2018) worked on superabsorbent polymers based on starch aldehydes and carboxymethyl cellulose. However, conventional SAPs based mostly on acrylic acid are non-biodegradable. Here, we developed ecologically benign and biodegradable polysaccharide based SAPs, of which carboxymethyl cellulose is the main component. Due to its non toxic and environmentally acceptable properties, the crosslinking agents that were chosen were

starch aldehydes and citric acid. The FTIR spectra analysis, aldehyde quantification and FE-SEM image morphology analysis of the periodate oxidized starch aldehydes were studied thoroughly. The cross linking mechanism was fueled by acetal bridges from the starch aldehydes to allow the absorption of significant amount of water into the SAP network. In particular, citric acid ester bridges blocked the access of water. The SAPs' swelling behavior was examined using the Schott's pseudosecond order kinetics model and Fickian diffusion model. The interaction between the swelling behavior and SAP morphology was characterized by FESEM pictures. With properly made polysaccharide based SAP's, a maximal equilibrium swelling ratio of 87.0 g g−1 was achieved. (6)

SAP can be extracted from bagasse of sugar cane prepared by Natthawut Neamjan et al. (2019). The end goal of this study is to make an environmentally desirable superabsorbent polymer (SAP) from sugarcane bagasse cellulose (SCB) to be used in the agricultural space, particularly horticulture and other agricultural applications for water resource management. SCB is polymerized using a rotating twin screw extruder. In this case, the water absorbing ability of polymer was increased by applying various ratios of AA and SCB. The results show that the swelling ability of ammountium persulfate/sodium sulfite initiator generated superabasorbly polymer is better than that of only ammountium persulfate. The enhanced SCB superabsorbent polymer was shown to improve the soil's ability to hold water when added to soil. The results suggest the potential usage of the superabsorbent polymer for improving agricultural management of the water resource. SCB and AA were co-rotated in twin screw extrusion with the aid of N, N methylene bisacrylamide as a cross linker and ammonium persulfate as an initiator, before their polymerization and then characterization was performed. (19)

One work of Negin Mahmoodi-Babolan et. al (2019) was bioinspired catecholamine/starch composites as superabsorbent for the restoration of the environment. A starch g(acrylic acid-co-acrylamide) superabsorbent was prepared by solution polymerization method, which may prove that starch based composite have potential qualities. Later, catecholamine functional groups were added to the pore surface of this absorbent by means of the oxidative polymerization of dopamine (DA). The adsorbent was optimized regarding the mass ratio of the monomers and synthesis conditions. Transmission electron microscopy (TEM) of PDA coatings and measurements

of the coating thickness yielded a value of 83 nanometers. The microspores of this bimodal mesoporous adsorbent had the specific surface area (SBET) of 2.8031 m2/g and swelling ratio of 5914.66%. Methylene blue (MB) was used as a model water pollution dye, and the adsorbent was then used to adsorb MB. When pH was set to nine, under the ideally designed conditions, the process happened in less than 100 min, and the maximum adsorption capacity was achieved at 2276 mg/g. Because of its exceptionally high adsorption capacity, this adsorbent has a lot of promise as a variety of environmental remediation applications. (20)

Jakub Misiewic et al. (2019) observed the properties of soil mixes and superabsorbent' AUL'. In the case of SAPs, the water is absorbed when it is loaded. It is examined in this study the absorbance under load (AUL) of a cross linked copolymer of acrylamide and potassium acrylate blended with coarse sand and sandy loam in ratios of 0.3%, 0.5% and 1.0%. In this case, 10, 20 and 40 cm of soil were applied as loads to these combinations. Results of AUL values exhibited the biggest disparities after 24 hours' comparison to control sample, especially at highest load of 5.9 kPa, which is equivalent to a topsoil load a depth of 40 cm. SAP addition of 1.0% to coarse sand and sandy loam resulted in AUL of 71.4 g/g and 52.7 g/g, i.e. 24.0% and 18.0% of the absorption in the control sample. In all tests the rate of water absorption was a sluggish one; collecting water that percolated down the surface of the soil is particularly important. Thus, these findings show that increasing the number of SAPs added results in decreasing water absorption by SAPs. (21)

By gamma irradiation, the superabsorbent Polymer glucomannan polyacrylate has been synthesized by Sri Mulijani et al. (2019) for the personal hygiene. Extremely popular research targets have been rapidly water or liquid absorption capable materials. One of several remarkable applications for which usage in diapers has attracted substantial interest is that their special characteristic allows them to be used. The superabsorbent polymer was made by combining solutions of glucomannan, potassium acrylic and acrylamide and cross linked using radiation (10 kGy). The superabsorbent polymer could swell up to 880 g/g of water, in just 15 minutes, proving just how massive superabsorbent materials are capable of getting. It also describes very good swelling characteristics in real urine with an absorption capacity of 110 g/g. Adding acrylamide, for instance, proved

to be a good choice for absorbent, thin diapers also because this added acrylamide was especially beneficial in actual pee, improving the swelling ratio. FTIR spectra analysis provided a validation of the finding in the above paragraph. In fact, the SEM pictures of the hydrogels also showed many holes (22).

Bacteria are the critical factors in medical concerns during wound healing, the antimicrobial effects are crucial in the development of a sustainable antibacterial superabsorbent hydrogel for individual medical application, according to Md. Obaidul Haque et al. (2019). By creating an ecofriendly hydrogel from cellulose with high absorption qualities and antibacterial properties it was possible for production of this hydrogel to be used in the personal healthcare industry. The hydrogel was prepared by simple free radical graft copolymerization using carboxymethyl cellulose (CMC), acrylamide and methyl methacrylate. This process was sped up by addition of N,N'methylene bis acrylamide as a cross linker and potassium persulfate as an initiator. Two different antimicrobial hydrogel versions were prepared by supplementing the hydrogel matrix with ZnO and TiO2, two antibacterial agents. It was found that hydrogels containing ZnO or TiO2 along not only perfectly retained the water absorption of 190 g/g and 173 g/g, respectively but also each had antibacterial activity averse to gram positive and gram negative microbes. The investigation also revealed that a decrease in the capacity to store water and an increase of antibacterial activity were induced by the greater number of antimicrobial agents. From the experimental results, it may be concluded that the synthesized hydrogel offers a promising breakthrough to improve problems in personal healthcare items. (23)

A. J. Capezza et al. (2019) probed possibilities for improving utilization using protein resources: organic, sustainable, absorbent materials. Apart from this, this viewpoint paper delivers a summary of present understanding of superabsorbent protein based materials (SAPs) and discusses about some functionalization techniques of these SAPs which have reported to make these SAPs more ecologically friendly. It serves to call attention to the variability of reactive groups in proteins, a property less common in other naturally occurring polymers. This variability, along with the lack of molecular changes and carefully chosen processing conditions must lead to other features and is then used to assess absorption capacity. Currently synthetic SAPs include superabsorbent gels that

contain proteins with water absorption of about 500 g/g and saline free swelling of about 12 g/g. Protein acylation is a particularly interesting method of functionalization that does not involve artificial monomers or polymers. This technique uses nontoxic chemicals to target lysine groups (-NH2) of the protein. Sustainable sources of protein for SAP manufacture include cheap, industrial byproducts such as potato protein and wheat gluten. The reasons for their suitability as dietary sources for upcoming bio based SAPs are twofold, they are vast available and, by comparison with the food industry, have low competition. A major problem, however, this relatively standard lysine content of these protein sources makes these protein sources more difficult by the reaction methods specified. Therefore, future research should aim to optimize the functionalization processes so as to obtain SAPs with the desired properties from these protein sources. Furthermore, methods such as increased saline free swell capacity (FSC), centrifuge retention capacity (CRC), and absorbance under load (AUL) should be included in a complete determination of the characteristics of protein based SAPs. In order to determine whether protein based SAPs can serve as viable, sustainable substitutes for the current petroleum based SAPs in use, considerable detailed and extensive life cycle analyses will need to be conducted. (24)

Capezza, A.J. et al. (2019) published a research paper on offering a simple and feasible method for converting potato protein concentrate (PPC), which is inexpensive, into powders that are biodegradable and incredibly absorbent. He tested five safe acylating agents for solvent-free acylation of the protein at varied reaction temperatures. The use of succinic anhydride at a temperature suitable for reaction at 140°C produced the best results. Under these circumstances, he was able to efficiently functionalize the material, resulting in the development of a useful network that enabled high fluid uptake with a minimum amount of material disintegration—specifically, protein hydrolysis—during the process. When compared to untreated potato protein concentrate, the SA-acylated potato protein concentrate exhibits significant improvement in its ability to swell in both water and salt by 600% and 60%, respectively. After being centrifuged at 1230 rpm for 3 minutes, the acylated potato protein also demonstrated an impressive blood swelling capacity of 530% and a saline liquid holding capacity of about 50%. This blood swelling capability is greater than 50% of what was shown in a super-absorbing substance with an

industrial fossil basis, which is frequently used in hygienic and medical items to absorb blood. These affordable protein-based acylated materials have outstanding swelling capabilities. They demonstrate their potential as SAP materials manufactured from industrial residues that are sustainable. These materials could find application in everyday hygiene items, which are currently primarily composed of fossil fuel-based SAPs. (25)

The superabsorbent hydrogel from carboxymethylated cassava starch was used by T. A. Afolabi (2019) for making sanitary napkins. A biodegradable superabsorbent hydrogel produced from using both native and carboxymethylated cassava starch was employed. First, the chemically modified starch was prepared by carboxymethylation from the cassava starch of cassava plant, and then the superabsorbent hydrogel was made from the modified starch. Analysis by FTIR spectra proved that carboxymethylated groups were present on the modified cassava starch granules, and X-ray diffractograms indicate that crystallinity is reduced after carboxymethylation. Studies using DSC

Results (Differential Scanning Calorimetry) indicated that hypermethylation decreased the temperature for gelatinization of the starch and its enthalpy of gelatinization. The superabsorbent hydrogel, which was made from the natural and carboxymethylated cassava starch had extremely large salt and water absorption capacities (> 100 times their dry weight) and the hydrogel made of carboxymethylated starch showed even greater capacity to absorb water. In addition, these hydrogels were very biodegradable with 70% degrading biologically in 14 days. Physicochemical properties of such superabsorbent hydrogels fashioned by natural and carboxymethylated cassava starch (CM cassava starch) have been shown to be similar to those of commercially available synthetic super absorbent hydrogels (e.g. casca, casahex), with the advantages of higher biodegradability. (26)

According to Chenhao Zhao et al. (2019), they modified starch with sulfamic acid such that the resulting sensitive-to-salt SAP held both nutrients and water well. However, the negative effects of electrolytes in solutions which greatly diminish the absorption capacity of superabsorbent polymers (SAPs) commonly prevent the use of such SAPs in absorbing liquids. This problem was overcome by creating a water absorbent polymer (polymerized from acrylic acid and sulfamic acid modified starch (Kalex 63 H) in a solution

polymerization process. In deionized water, it also showed remarkable swollen proportion of 1026 gg-1, while 245 gg-1 was observed in a 0.9% sodium chloride solution. When compared to typical starch grafted acrylic SAPs, these numbers reflected a stunning increase of 99.5% and 13.4%, respectively. Simulated leaching tests in a soil column were also carried out to assess water and fertilizer retention. The findings demonstrated that the rising water absorption capacities in salt solutions were due to the sulfonic acid groups' ability to adsorb and assist the transfer of water molecules. Notably, compared to scenarios without superabsorbent treatment, the treatment capable of withstanding salt superabsorbent greatly decreased water at 18.5%, nitrate at 22.8%, ammonium nitrogen at 88.0%, and water-soluble potassium at 63.8%. This study's methodology has the potential to direct the creation and broad use of salt-tolerant SAPs in horticultural and agricultural applications. (27)

Qianyu Zhang et al. (2020) made a superabsorbent polymer by employing rice starch. This study set out to develop brand-new, eco-friendly superabsorbent materials (SPAMs) based on rice starch because indica rice starch has a comparatively high amylose concentration (23.6%), it has a greater ability to absorb water (439 g/g) compared to other established SPAMs. Through hydrogen bonding, the amylose concentration improves the relationship and arrangement of the stability of polymer chains. Due to the stiffness and composition of the branching amylopectin chain, glutinous rice starch-based SPAMs with a high amylopectin content (75.1%) showed superior water absorption capabilities (399 g water/g). After three consecutive reuse cycles, the reswelling capacity of indica rice starch-based SPAMs was maintained at 70%. The order of the cations' effects on SPAM's water absorption was $K^{+} < Na^{+} < Ca^{2+} < Al^{3+}$ (28)

El ˙zbieta Czarnecka et al. (2020) proposed a method to prepare semi-natural super absorbents based on starch-g-poly (acrylic acid). She presented that It was determined how well the hydrogels absorbed water in distilled water with a solution of 0.9% NaCl. These hydrogels' ability to swell is determined by the monomer ratio and cross-linker (N, N'-methylene bisacrylamide) concentration. N, N'-methylene bisacrylamide concentration improves with a decrease in swelling. The effects of charge screening and ion cross-linking for monovalent and multivalent cations, respectively, can be utilized to clarify why salt solutions absorb less water than purified water. The outcomes obtained

are consistent with the idea that faster liquid absorption is caused by smaller particle sizes. (29)

Giovanni F. de Lima et al. (2020) researched using nano-cellulose as an augmentation in hydrogels made of carboxymethylcellulose that are extremely absorbent. Carboxymethylcellulose (CMC) is a generally available and reasonably priced substance that may be used to create superabsorbent hydrogels that are beneficial to the environment and have a variety of uses. Although different fillers can be applied to enhance the mechanical characteristics of these hydrogels, they occasionally have the potential to reduce swelling. A great option to improve the physicochemical qualities of hydrogels is nitrocellulose (NC). Using citric acid as a cross-linker and NC as a filler in three different concentrations—1%, 3%, and 5% by weight—they created CMC-based hydrogels for this investigation. The cross-linking reaction's occurrence was verified using Fourier Transformed Infrared Spectroscopy (FTIR), and the hydrogel's expansion was confirmed by the appearance of an elevation connected to the carboxylic acid functional group.
Remarkably, even though both materials had a similar structure, the addition of NC had little impact on the FTIR spectrum. Each of the hydrogels created for this study showed evidence of being superabsorbent, with an ability to absorb more than 40 gg^{-1} of hydrogel, according to analysis of water absorption. Notably, the addition of 3% of NC significantly increased the water absorption capacity, producing a material that swelled to a degree of about 100 grams of water per gram of hydrogel. NCs worked well as fillers, significantly raising the storage modulus and raising the material's mechanical strength because of this. The hydrogels created in this investigation had extraordinary qualities that made them appropriate for a range of uses. (30)

In 2021, Dipankar Das et al. categorise and fabricate superabsorbent hydrogels based on cellulose for agriculture applications. Most hydrogels commonly being used in agricultural applications are in fact not biodegradable, thus it is important to first remember this before discussing the fact that they facilitate carbon capture. Thus, the hydroxyethyl cellulose (HEC) and carboxymethylcellulose sodium salt (CMCNa) hydrogels, producing and evaluating the cross link with citric acid (CA) are the aim of this work. With this in mind, Fourier-transform infrared spectroscopy, method is used to chemically analyze the synthesized hydrogels. Furthermore, hydrogel matrix, together

with acid hydrolysis-produced cellulose nanocrystals (CNCs) were also added into the hydrogel matrix and their effect in the characteristics of the material was assessed. In their investigation of CA cross-linker effects on hydrogel swelling capacities, about 600% was observed to be a significant swelling capability of the hydrogel that was produced with a 2% CA cross link concentration. However, it is worth noting that the tensile effectiveness has declined due to the addition of CNCs as reinforcement agents. This is clearly shown in images taken with scanning electron microscope (SEM). To optimize the outcomes out of hydrogel system, the usage of the crosslinkers is carefully balanced and the CNCs are correctly distributed in hydrogel matrix. It could allow the production of hydrogel polymers with designed water absorption and release characteristic, and therefore the better utilization of water resources in Agricultural applications. (31)

A new method for the formation of superabsorbent polymers was introduced in 2021 by Nahed A. Abd El Ghanyet et al. where corn starch is applied. Swelling capacity measurement revealed that the swelling capacity of all hydrogels in saline solutions, as well as in acidic and alkaline buffer solution, was much greater than that in a neutral solution. This is so because their framework moves with the ions that move through it. The variation of swelling with the cross-linking density increased. The hydrogels were found to retain their stability for at least 24 hours upon exposure to degradation testing at pH buffer mixtures of 4, 7, and 9, and after 48 hours none was found degraded. When compared to native starch, the results of the study which evaluated thermal stability of starch hydrogels and their swelling in water and a 9% saline solution revealed high thermal stability and ability to swell to a great extent in water and 9% saline solution. In addition, swelling in buffer solutions that are neutral, alkaline, and acidic tended to be greater in them. As it is, after 96 hours, it is noteworthy that less degradation was seen in acidic and alkaline environments. (32)

Polyacrylic acid were used to develop, prepare, characterize and expansion properties of superabsorbent polymer composites by Marzieh Jafari et al. (2021). Through an aqueous solution polymerization procedure comprising blends of polyvinyl alcohol (PVA)/acrylic acid (AA) and carboxymethyl cellulose (CMC)/CMC, with the initiation provided by benzoyl peroxide, superabsorbent polymers (SAPs) were produced. These SAPs were produced under simple conditions and with a straightforward approach. The

biodegradable superabsorbent CMC/AA copolymer and the PVA/AA copolymer were compared. The super absorbent, environmentally friendly composite composed of CMC and AA showed the greatest potential for swelling. With a larger benzoyl peroxide content, longer immersion times, and higher reaction temperatures during polymerization, SAPs' capacity to absorb water improved. As the quantity of the cross-linking agent N, N'-methylene bisacrylamide increased, the gel fraction of SAPs dropped. (33)

Seyed Rahman Djafari Petroudy et al. (2021) prepared sustainable superabsorbent materials made from wheat straw that have been with electrospun cellulose nanofibers. Superabsorbent polymers are currently used in the healthcare and agriculture industries, which raises substantial societal problems because they are predominantly made from petroleum-based ingredients. In this case, electro spun cellulose nanofibers (ECNFs) were used to create superabsorbent fibers (SAFs). TEMPO-mediated oxidation served as a pretreatment step after the cellulose from wheat straw was extracted. The cellulose was subsequently dissolved in trifluoroacetic acid to create ECNFs using the electrospinning method. The highest swelling ratios were attained by the ESAFs (Electro spun Cellulose Nanofiber-based Superabsorbent Fibers), which were made of oxidized ECNFs containing 15% poly (sodium acrylate) and measured at 225 g/g in purified water as well as 208 gg^{-1} in brine solution made of 0.9% by weight. According to the FESEM research, ESAFs created strong three-dimensional architectural networks. It was also noticed that these ECNFs had low ionic sensitivity. The prepared ESAFs provide a green and sustainable alternative for a variety of applications, which helps reduce the use of plastic microspheres. (2)

Mei Chuen Peng et al. (2021) worked on the performance evaluation of chia seeds, chia flour, and mimosa pudica hydrogel for sanitation towels made of polysaccharide-based superabsorbent polymers. Antimicrobial and biodegradable experiments done on MPH indicate that they may be antimicrobial and biodegradable. Among all used seeds, MPH appears to be a potential SAP for usage in sanitary napkins, when compared to chia seeds and chia flour. (4)

A breakthrough ecologically friendly and sustainable absorbent material is superabsorbent polymer (SAP) made from pearl millet starch. The starch is then extracted from the millet grains using the process by a sodium hydroxide solution. Instead, this process produces a

starch markedly different from that from traditional source having special properties. Superabsorbent polymers are well made from millet starch because of the intrinsic properties of millet starch. Commercially, due to their high swelling capacity and application range, the high use of millet starch would transform to a super absorbent polymer for storing and release water. It was stable, biocompattible and can make up environmental problems associated with applying the conventional non-biodegradable superabsorbent polymers. (4)

Elzbieta Czarnecka et al. (2021) introduced a method to create and characterize superabsorbent polymers. Chemicals used in this procedure included potato starch, acrylic acid, acrylamide, poly(vinyl alcohol), 2-hydroxyethyl methacrylate, and 2-acrylamido-2methylpropane sulfonic acid, with ammonium persulfate and potassium persulfate helping with the initiation process. As the Na^+ ion concentration rose, the substances' ability to swell decreased. This demonstrates that the charged groups' ionic repulsion injected through the matrix of gel through external pH modification is most likely the major element determining the observed swelling behavior. The order of absorbance was determined when the parameter relating to the absorption rate of deionized water was examined soluble starch-g-poly (acrylic acid-co-2-hydroxyethylmethacrylate > starch + PVA interpenetrating polymer networks) > super absorbent polymer. Saline solutions showed the same order of lower absorbance as well. (34)

Md. Moynul Hassan Shibly et al. (2021) worked on the creation of a menstruation pad made of biological polymers and the standard testing against products sold in stores. Women and girls in the least developed nations face a big problem when it comes to menstrual hygiene issues. Menstruation management practices frequently employ unsanitary materials, which can cause several health problems. Although sanitary napkins are widely available locally and offer excellent absorbance and flexibility, many of them include synthetic superabsorbent polymers (SAP) in their core layers, which are neither skin-friendly nor biodegradable. This study seeks to replace SAP with an eco-friendly biopolymer to create a sanitary and biodegradable sanitary pad. The objective is to offer rural women a dependable, environmentally friendly menstruation hygiene option. He looked at a few of the current sanitary pads on the market in our area to develop this type. For the core absorbent layer, he created six distinct models employing many biopolymers,

including cotton, viscose, wood pulp, sodium alginate, and carboxymethyl cellulose (CMC), in various combinations. To standardize the sanitized pads' fundamental qualities and worth, he undertook several evaluations, including tests for antibacterial activity, wicking capability, and water retention. According to the experimental results, utilizing sodium alginate and CMC as SAP substitutes produces the best outcomes that are equivalent, particularly when sodium alginate, CMC, and cellulosic fiber are combined in the template. Furthermore, these findings show that this newly created model has significantly improved in both design and cost-effectiveness. Sanitary napkins devoid of SAP might offer a secure and eco-friendly disposal option. (35)

Hae Chan Kim et al. (2022) worked on new itaconic acid-based SAP composites utilizing oxidized starch. SAP based on itaconic acid made from biomass must improve its gel strength and absorption capabilities to be commercially viable. By in-situ aqueous solution copolymerizing poly (itaconic acid-co acrylic acid) SAP composites using oxidized starch (OS) as a filler, he created novel materials. By assessing free absorbance, centrifuge retention capacity, and absorbance under load in a saline solution, we further evaluated the absorption capabilities of the SAP composites with various OS levels, cross linker quantities (1, 6-hexanediol diacrylate), and degrees of monomer neutralization. After eliminating unreacted monomers and oligomers, he calculated the gel content and used scanning electron microscopy and BET analysis to look at their morphologies. Finally, he contrasted the ideal outcomes produced by the response surface methodology (RSM). As a result, he determined the ideal polymerization conditions for the 85 g of polymer SAP composites, which included 4.5 g of OS, 2.0 g of cross-linker, and a 75% degree of neutralization. With a centrifuge retention capacity (CRC) of 55.7 g/g and an absorbance under load (AUL) of 10.2 g/g, these ideal SAP composites showed exceptional performance. (36)

Ariel V. Melendres et al. (2022) studied the impact of the starch from maize on superabsorbent polymers' biological degradation and absorption. Free swell capacity (FSC) testing was done while a 0.9% NaCl solution was present. The superabsorbent polymer (SAP) was subjected to the Gel Layer Permeability (GLP) test, in which samples were left in a 0.9% sodium chloride solution for an hour to partially swell. The outcomes of the FSC and GLP tests revealed that the treatment with maize starch altered the

permeability and absorption properties of the SAP. This alteration was credited to the treatment's increased gel strength. (37)

Muddassir Ali Memon et al. (2022) fabricated a highly absorbent polymer composite based on nanoclay for applications requiring water absorption. The mechanical, chemical, and physical qualities of materials are improved by adding naturally occurring fillers like clay and other minerals. In this work, he demonstrated the graft copolymerization of acrylic acid and acrylamide to create a nano clay-based superabsorbent polymer. This study primarily examines the effects of filler (nano-clay), cross-linking agent, and initiator concentrations on the copolymer's absorption characteristics. Six distinct examples, precisely 30, 32, 34, 36, 38, and 40 wt% of nanoclay were created. The polymer matrix included these nanoclay percentages. With immersion periods ranging from 2 to 24 hours, a time-dependent immersion experiment was carried out using tap water, deionized water, and brine. It is important to note that specimens with 38 and 40% nanoclay showed a reduction in absorption ability. This decline may be related to the polymer matrix's excessive development of physical and chemical cross-linking networks. Additionally, a decrease in absorbance was seen when these superabsorbent polymer composites were suspended in brine. Specimens containing 36-weight percent nanoclay likewise showed this pattern after 4, 6, and 24 hours of immersion. The polymer composite that was complemented by 38 weight percent nanoclay showed better consistency, withstanding temperatures as high as 350°C, according to thermal stability studies. (38)

Peenal Arvind Mistry et al. (2023) prepared applications of chitosan superabsorbent biological polymers in sanitation and healthcare. Consumption of sanitary items and diapers has been rising steadily. The usage of chemical-based sanitary products has given rise to several issues, including their sluggish decomposition, which makes them harmful to the environment, as well as their propensity to lead to bacterial infections and nappy rash in infants. As a result, the use of natural sanitary products is becoming more popular because of their biodegradability, lack of toxicity, and compatibility with the human body. The production of superabsorbent materials is the subject of numerous research projects that include natural polymers like cellulose, starch, alginate, and xanthan gum. One such natural polymer is chitosan (CS), whose structural functional groups are responsible for its antibacterial capabilities. Additionally, CS is widely accessible, biodegradable, and

safe. The features of CS and various techniques for producing superabsorbent items using natural polymers, such as pads for sanitary use and diapers, are the main topics of this paper. It also mentions in passing the various uses of chitosan as a biopolymer in fields like cuisine, textiles, cosmetics along medicine. This study also recommends chitosan as a favorable choice in the production of feminine hygiene products for both women and children. Due to its extraordinary capacity for retaining water, swelling, and antibacterial properties, CS stands out as a strong choice for the creation of superabsorbent biopolymers. (39)

CHAPTER 3

METHODOLOGY

3.1 Required chemicals

Pearl millet grains (purchased from a local market in Lahore), Acrylic acid (AA), Acrylamide (AM), N-N' Methylene bisacrylamide (MBA), Potassium persulphate (KPS), Sodium hydroxide (NaOH), Nitrogen gas, Tetramethyl ethylene diamine, Methanol, and distilled water were obtained from the lab store of Superior University.

3.2 Preparation of pearl millet starch

Medium-sized pearl millet grains were purchased from the market. They were sifted through a sieve to eliminate unwanted husks and dust. These sifted pearl millet grains were placed in the beaker, and water was added to completely rinse seeds until all dust was gone. They were rinsed two times more like this. Then, we continued to dry these pearl millet grains for 30 minutes in an oven that had been preheated to 45°C. A 0.25% NaOH solution was prepared in a volumetric flask in distilled water. 70 grams of pearl millet grains were soaked in 400 milliliters of a 0.25% NaOH solution for one day at ambient temperature. The mixture is then ground with a mortar and pestle. After that, 400 milliliters of the exact 0.25% NaOH solution were agitated and left to sit for the night. After that, we drained the liquid and used a mixture to combine the other ingredients. Then, pass it through a 40-mesh sieve and give it a thorough water rinse.

Once more, the liquid was wasted after blending the material in small amounts with 0.25% NaOH in a mixture machine. It was passed through a 40-mesh sieve, and we gave it a thorough water rinse. A mortar and pestle were used to pulverize the mixture one more time. It was passed through a 40-mesh sieve again and given a thorough water rinse. A Mortar and pestle were again used to pulverize the mixture one more time. It was put through a 40-mesh sieve again and given a thorough water rinse. Additionally, we passed it through a 200-mesh sieve and gave it a water rinse.

Finally, we centrifuged it to separate the gluten from the starch after passing it through a 270-mesh sieve. Use small amounts of water to carry out this procedure numerous times,

if necessary. Whatman No. 2 was used to filter the final product. Finally, let it dry at 45°C in a convection oven. 45.5 g of starch was recovered from 70 grams of pearl millet grains.

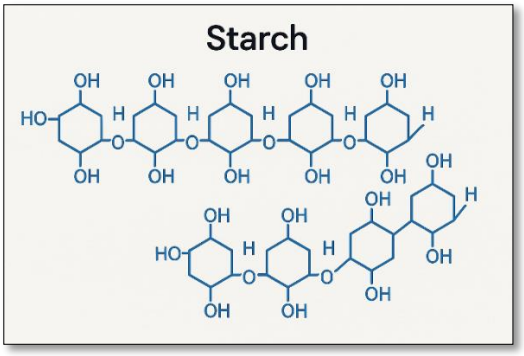


Figure 3.1 Structure of starch

3.3 Preparation of PMSSAP

The reaction was conducted in a flat-bottomed borosilicate glass flask equipped with an upper stirrer, a temperature sensor, and a nitrogen intake. The flask was first filled with 6 grams of starch and 100 mL of distilled water. Next, nitrogen gas was introduced to the flask to generate an inert atmosphere. After increasing the temperature for fifteen minutes, hold it at 85°C to allow the starch to gelatinize.

After that, the reaction system was well mixed, and the temperature was decreased to 40°C before the monomers (acrylic acid and acrylamide), cross-linked (methylene bis acrylamide), and initiator (potassium persulfate) were added. The reaction continued at 40°C for a further 2 hours at this rate when the activator (tetramethyl ethylene diamine) was inserted.

Methanol was added to cease the reaction, which resulted in the precipitation of the end product. The precipitate was subsequently put through filtering. An ethanol-water combination (80:20) was used to remove any homopolymers from the grafted polymer that may have been generated during the reaction after they had been properly washed, usually three times. The sample was then eliminated by drying at 55°C. Hence, superabsorbent polymers from pearl millet starch have been prepared.

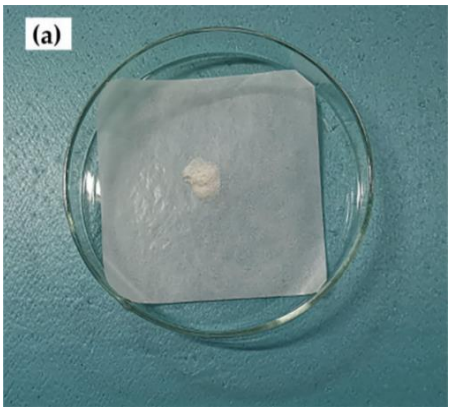


Figure 3.2 Dry PMSSAP

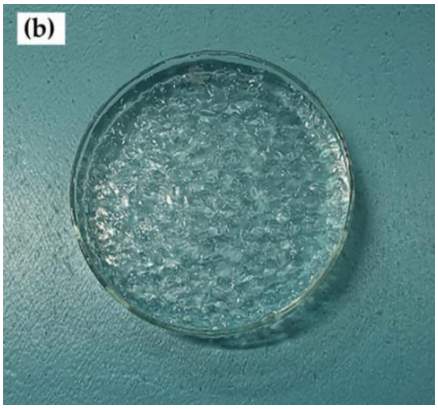


Figure 3.3 Water absorbed PMSSAP

3.4 Characterization of PMSSAP

3.4.1 Functional group analysis of PMSSAP

PMSSAP was submitted in the form of white powder to analyze the FTIR spectra including identification of chemicals and material characteristics.

3.4.2 Topographical analysis of PMSSAP

PMSSAP sample was dried in an auto-desiccator for 24 hours before analysis and dispatched for SEM analysis.

3.4.3 Thermogravimetric (TGA) analysis of PMSSAP

PMSSAP (in powder form) was subjected to thermogravimetric analysis (TGA) to determine the thermal stability.

3.4.4 Structural analysis of PMSSAP

PMSSAP sample was dispatched in the form of finely ground to check the extent of crystallinity.

3.4.5 Absorbance capability of PMSSAP

Using gravimetric measurement, the PMSSAP's water absorption capability was evaluated. 1 gram of the sample was submerging in 100 milliliters of distilled water at

room temperature for a duration of 1 hour. After that, an 80-mesh nylon bag was used to filter the expanded sample to remove any remaining unabsorbed water, and this process was repeated until the sample no longer leaked free water.
The above formula was used to determine the PMSSAP's water absorbance.

$$Q_{eq}\ (g/g) = W_2\text{-}W_1/W_1$$

The mass weights of the dry and swollen samples (g), respectively, and W_1 and W_2

3.4.6 Determination of PH

pH of PMSSAP was detected by mixing 1g of the sample with 100 mL while the mixture was continuously mixed for ten minutes. After mixing for 60 seconds, the pH was measured by placing a pH electrode in the supernatant solution. PMSSAP has a pH of 8.3 at ambient temperature.

CHAPTER 4

RESULTS

4.1 Pearl millet starch analysis

Pearl millet was used for obtaining starch by employing a 0.25% NaOH solution, yielding a clean starch concentration of 45.5 g per 70 g of pearl millet grain. The humidity level of the starch was 10.6%. Pearl millet starch exhibits greater compatibility for the preparation of PMSSAP because of a larger concentration of amylopectin, which is recognized for its enhanced solubility. This conclusion is reinforced by the increased swelling power of pearl millet starch.

4.2 PMSSAP

After being synthesized, the PMSSAP took on the consistency of a gel, which was then dried for two hours at 55°C to remove any extraneous volatile organic compounds and moisture. Despite the prolonged drying time, the finished product had the consistency of a white and crystalline powder. The product has considerable promise as a novel biodegradable PMSSAP.

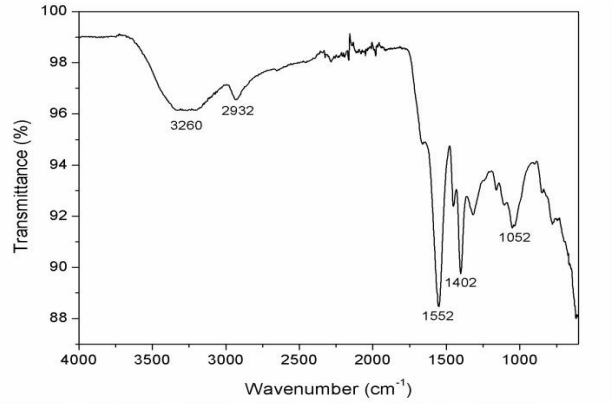
4.3 Characterizations

4.3.1 Functional group analysis of PMSSAP

PMSSAP was characterized by using infrared spectroscopy to pinpoint the precise kinds of chemical bonds that were present in it. According to the resulting spectra, it typically showed absorption peaks that coincided with the functional groups predicted. The stretching vibration of hydrogen bonds involving -OH groups is shown (Fig. 4.1) by a strong and wide absorption band at 3260 cm^{-1}. Furthermore, a medium-intensity absorption signal at 2932 cm^{-1} denotes aliphatic structures' symmetric and asymmetric CH stretching vibrations.

Furthermore, the bending vibration of C-H bonds in methyl groups is responsible for the peak at 1402 cm^{-1}. The existence of -C=O bonds, which are connected to conjugated

ketones, carboxylic acids, and esters, may be seen in the absorption bands at 1552 and 1052 cm^{-1}, respectively. The discovery of these several functional groups indicates that the PMSSAP used in this work has the potential to be an effective product.



4.1 Functional group analysis of PMSSAP

4.3.2 Topographical analysis of PMSSAP

PMSSAP showed microstructure morphology in determining how well PMSSAPs can absorb water. PMSSAP has a better, more consistent, more porous structure, which improves its capacity to absorb water. The hydrophilic groups of the components of the PMSSAP interacted with one another in these pores. During the PMSSAP synthesis process, water evaporation caused the creation of these holes.

According to the SEM examination, PMSSAP has an average diameter of 200 nm, which is in line with findings from their studies of polysaccharide-based polymers. Particle sizes of about 200 nm are shown in the Millet starch-based polymers Scanning Electron Micro. It also reported similar results from SEM analyses, showing a uniform morphology and irregular pores with a diameter of 5 to 1000μm that were apparent at lower magnifications. Furthermore, surface studies at lower magnifications show the presence of CH particles and millet fibers on the PMSSAP surface, with lengths ranging from 5 to 500μm. (Fig 4.2)

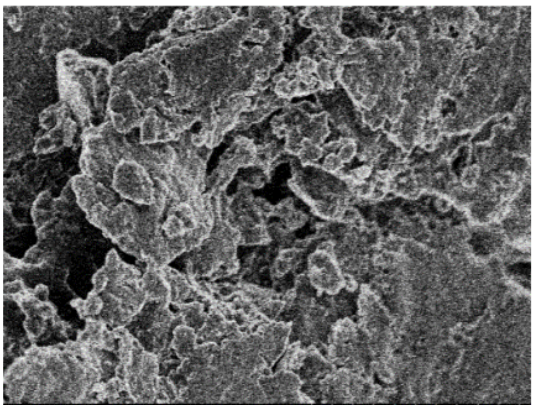


Figure 4.2 Topographical analysis of PMSSAP

4.3.3 Thermogravimetric analysis (TGA) of PMSSAP

Thermogravimetric analysis (TGA) is the key and significant physical property of a superabsorbent polymer. At 133 °C, starch lost 10% of its weight due to moisture evaporation. It had strong thermal resilience up to 300°C, losing a total of 20% of its weight. Over this temperature range, a noticeable weight loss started to happen and reached 50% in the 300– 335°C range, which indicates starch molecule breakdown. The rate of weight loss reduced as the temperature rose, reaching a maximum loss of 76.1% at 600°C.

As opposed to its untreated counterpart, treated starch showed improved thermal stability. The thermogram of the treated starch showed a progressive breakdown across the full temperature range without strong inflections.

A weight loss of 76.1% was seen up to 600°C, indicating increased thermal stability. The creation of a cross-linked 3-D structure and chemical alterations brought on by grafting and cross-linking with vinyl monomers may be responsible for this enhancement. Fig 4.3

TGA (Thermogravimetric Analysis) graph showed analysis of thermal stability and its composition of a material. Weight changes as function of temperature for the sample were shown during heating to show decomposition steps, moisture content, and thermal resistance. Stability indicated material; sharp dropped (i.e. down sloped regions) indicated events such as moisture evaporation or material breakdown. Degradation temperatures were determined based on the onset and the end of the weight loss steps.

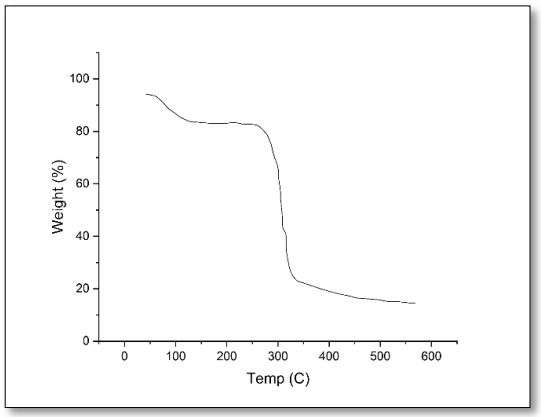


Figure 4.3 Thermogravimetric analysis of PMSSAP

4.3.4 Structural analysis of PMSSAP

The spectra of the millet starch and modified starch samples showed different characteristics. Patterns found in grain starches, strong diffraction peaks at 15.61,17.83, 18.14, and 23.79° (2θ) were present in millet starch in its natural condition, when its relative crystallinity was 21.01%. However, the intensity peak of millet starch evolved into a larger, more expansive peak with 6.89% crystallinity.

The XRD results suggest that the starch structure is partially transitioning from crystalline to amorphous. This may have happened due to both gelatinization of the starch and reaction of starch with vinyl monomers and random crosslinking between the polymer.

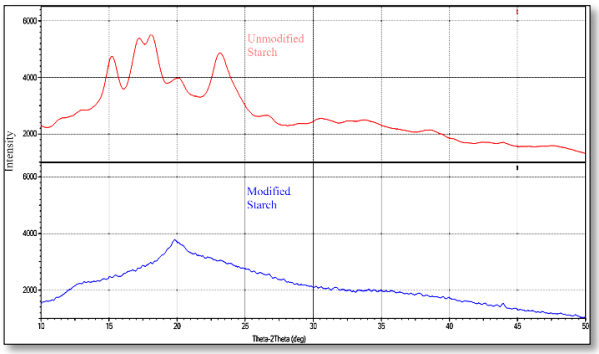


Figure 4.4 Structural analysis of PMSSAP

4.4 Physio-chemical measurements

4.4.2 Water absorbance capability of PMSSAP

4.4.2.1 Effect of pearl millet starch on water absorbance

With the presence of several hydroxyls (–OH) groups along its glucose polymer chains, starch is very hydrophilic. Hydroxyl groups of starch readily form hydrogen bonds with water molecules and thus starch is able to absorb water very efficiently. If starch is mixed with water, it swells and can form gels or pastes by lowering the temperature and concentration. The contents of acrylic acid (AA), the amounts of N, N'-methylene bisacrylamide (MBA), KPS and acrylamide were set at 98.5, 0.05and 11.34 weight percent, respectively at room temperature. As the starch ratio increased, the water absorbance increased steadily; however, beyond a certain point, the water absorbance sharply decreased as the monomer weight increased further. This behavior can be explained by the fact that as the concentration of monomers grew, the reaction intensified and hydrophilic functional groups like -OH, -COOH, and -COONa accumulated.

It has been reported that -COONa ionizes to form Na^+ and $-COO^-$. The osmotic actions and ionic hydrophilia of $-COO^-$ help to gradually increase the polymer's water absorbance. Moreover, the reaction rate was accelerated as the concentration of monomers rose because they were transported closer to the polymer backbone.

However, the polymer's water absorbance decreased as soon as the monomer concentration above an ideal threshold. This decrease could be explained by the system's increased viscosity brought on by the higher monomer content, which made it more difficult for macro-radicals and monomer molecules to move around. Furthermore, the probability of chain transfers to monomer molecules increased as homopolymerization surpassed graft copolymerization.

The graph shows how the amount of millet starch affects PMSSAP's water absorbance. 6 grams of millet starch produced the highest absorbance value (Qeq) while thirty grams of millet starch produced the lowest absorbance value (Qeq). 6 grams of starch gave high water absorption of 320.47s g/g whereas lowest absorption of 80 was seen at 8 wt percent.

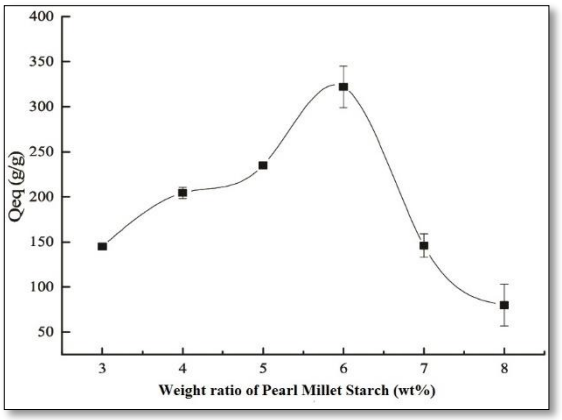


Figure 4.5 Effect of millet starch on water absorbance

4.4.2.2 Effect of initiator on water absorbance

KPS (potassium persulfate) content also affects the PMSSAP's water absorbance. In this study, KPS concentrations ranged from 1 to 5 content percent. In addition, millet starch, acrylamide (AM), acrylic acid (AA), and N, N'methylene bisacrylamide (MBA) were utilized in the manufacture of the PMSSAP at room temperature. Based on KPS-free measurements, these amounts were 6, 11.5, 98.5 and 0.05 weight percent, respectively.
The graph shows how the initiator content affects the polymer water absorbance. It illustrates how the water absorbance of the polymer rose in tandem with the initiator content, peaking at the ideal initiator-to-monomer ratio. The water absorbance started to decline after this. The initiator is crucial in defining the molecular weight of the polymer as well as the rate of polymerization in free-radical polymerization. Insufficient radical production impeded the development of an effective polymer network at low initiator and radical concentrations. The absorbance at low initiator ratio was 240 g/g at 5 wt percent of KPS. But when the initiator content rose, the radicals' buildup made it easier for the polymer network structure to expand, which improved its water absorption. The highest absorption was seen 325 g/g at 4 weight percent of KPS.

An excessive amount of radicals was produced in the system as the initiator concentration increased. Because of the increased chain termination events caused by this overabundance, the polymer's molecular weight decreased, resulting in shorter average macromolecular chains and less free volume accessible inside the superabsorbent polymer

matrix. As a result, the polymer chains broke off early, and the reaction was completed faster. As a result, the polymer's water absorbance eventually decreased since the initiator content was higher than it should have been.

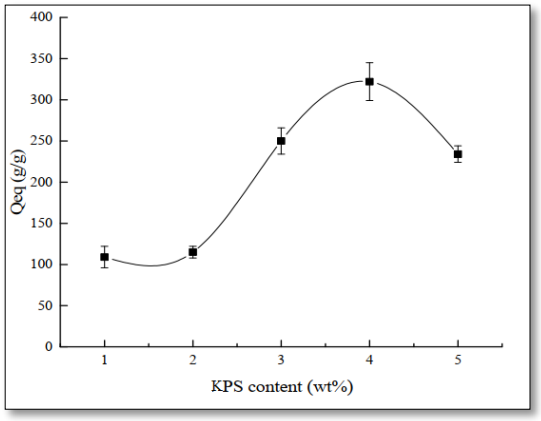


Figure 4.6 Effect of initiator on water absorbance

4.4.2.3 Effect of cross-linker on water absorbance

Cross-linker content also affects the PMSSAP's water absorbance. The N, N'-methylene bisacrylamide (MBA) concentration was adjusted between 0.0 and 0.4 content percent. These samples' reaction conditions were as follows: 6, 11.5, 98.5, and 4 weight percent, respectively, of millet starch, acrylamide (AM), acrylic acid (AA), and potassium persulfate (KPS), based on measurements excluding MBA, were obtained at room temperature. By joining linear polymer chains, the crosslinker creates a three-dimensional network structure by acting as a molecular bridge. Since even little changes in crosslinker density can significantly change the characteristics of the superabsorbent polymer, the density of the crosslinker plays a crucial role in regulating absorbance behavior and the copolymerization reaction. The graph shows how the amount of crosslinker affects water absorbance. It is clear that the polymer's water absorbance dropped as the crosslinker content rose. In order to create a network that can absorb and hold onto water molecules from the aqueous solution, the crosslinker adds a great quantity of crosslinking spots to the polymer chains during the reaction.

But too much crosslinker results in too many crosslinking spots, which raises the polymer network's crosslinking density. This causes an excessively dense network to grow, which

limits the amount of space available for water absorption and, eventually, lowers the polymer's water absorbance. Essentially, a larger number of crosslinking spots results in an excess network structure that limits the polymer's capacity to swell and efficiently absorb water. Highest absorbance of 345 g/g was recorded at 0.05 wt percent whereas lowest absorbance of 50 g/g was recorded at 0.4 wt percent.

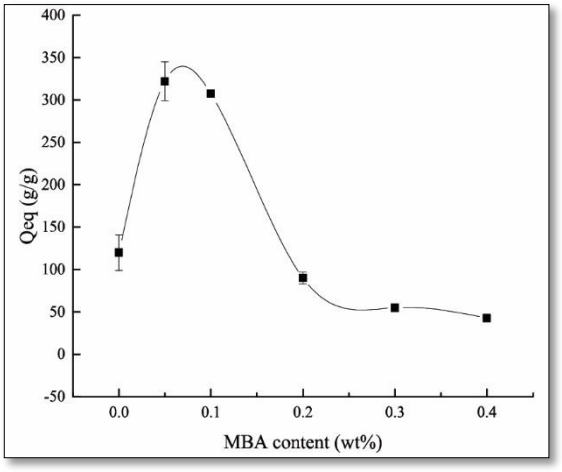


Figure 4.7 Effect of MBA on water absorbance

4.4.2.4 Effect of salts on water absorbance

Salt samples like NaCl and KCl solutions at various concentrations showed the impact of different cations on the superabsorbent polymer's capacity to absorb as shown in graph. The network of the PMSSAP shrinks when it is submerged in electrolyte solutions, which causes a steady decline in absorbance capacity as the concentration of the salt solution increases. The kind and quantity of ions in the solution have a big impact on how the PMSSAP behaves as it swells. Highest absorbance was seen at 330g/g but as the concentration of salts increases then lowest absorbance was recorded 50 g/g at 1.2 wt percent. It had been established that the PMSSAP absorbed less in salt solutions than it did in deionized water. As the concentration of the salt solution rose, it was discovered that submerging the PMSSAP in electrolyte solutions led the network to compress, reducing the swelling capacity gradually. There are two are two main reasons for lowering of water absorbance like screening effect of ions such as sodium ion and osmotic pressures surrounding ionized water of superabsorbent polymer. As a result, the PMSSAP showed

considerable water absorption when submerged in pure water, which contains no salt. However, even trace levels of salt in the system dramatically reduced its capacity to absorb water.

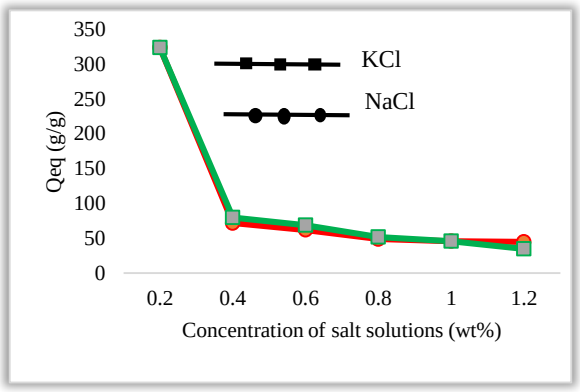


Figure 4.8 Effect of salts on water absorbance

4.4.2.5 Effect of temperature on water absorbance

Absorbency tests at elevated temperatures from 55°C and above were performed to evaluate the thermal stability and to study the practical performance of the synthesized starch based superabsorbent polymer (SAP). Such testing is necessary since SAPs are under usage in the environment where temperature fluctuations take place, for instance, in agriculture (e.g. soil exposed to sun), hygiene products, packaging. The absorbance behavior can be affected by increased kinetic energy of water molecules, possible dehydration of the hydrogel, or thermal collapse of the polymer network, under elevated temperatures. Testing at higher temperatures, therefore, is used to determine how much absorption capacity is present, whether the network is intact enough, and if the material can be used for real world use under thermal stress. It is evident how temperature affects PMSSAP by submerging the sample in water solutions at various temperatures, including 55°C, 60°C, 65°C, 70°C and 75°C, to determine the effect of temperature on absorbance.

As the reaction temperature increased, the polymer's water absorbance first climbed and subsequently fell. According to research, the crosslinking efficiency declined as the reaction temperature rose within a lower temperature range. Water absorbance increased as a result of the decrease in crosslinking efficiency at a constant crosslinker level. The

highest absorbance was noted at 337 g/g. Furthermore, the rate at which acrylic acid (AA) diffused to the macroradicals hiked with temperature. Moreover, the probability of bimolecular collisions between initiator and starch molecules rose in tandem with the reaction temperature. The quantity of polymer macroradicals increased as a result of these collisions, hastening the start and expansion of the polymer chains.

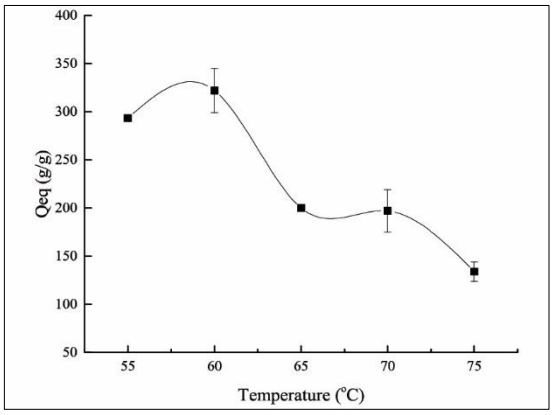


Figure 4.9 Effect of temperature on water absorbance

4.4.2.6 Effect of pH on water absorbance

The pH value has a significant impact on the polymer's absorbent ability along with the kind and concentration of ions; this is a crucial factor in practical applications. The absorption capacity of superabsorbent polymers was assessed in solutions with pH values ranging from 3.5 to 12.0. The polymer absorbance grew from 3.5 to 6.0, then decreased at 7.0, then increased again at 8.0, and eventually continued to decrease, as seen in the graph. While pH 9.0 displayed the second-highest absorption, pH 8.0 displayed the highest absorbance. While pH 9.0 displayed the second-highest absorption, pH 8.0 displayed the highest absorbance. At higher pH values (>4.6), the presence of COO^- groups caused strong electrostatic repulsion that encouraged the ionization of carboxylic acid groups, increasing swelling capacity.

However, as the pH increased to 7.0, most of the basic and acidic groups stayed non-ionized. Electrostatic repulsion between the COO^- groups caused the network to grow. However, as alkalinity increased, water absorption decreased due to the screening effect of Na^+ counterions in the swelling medium.

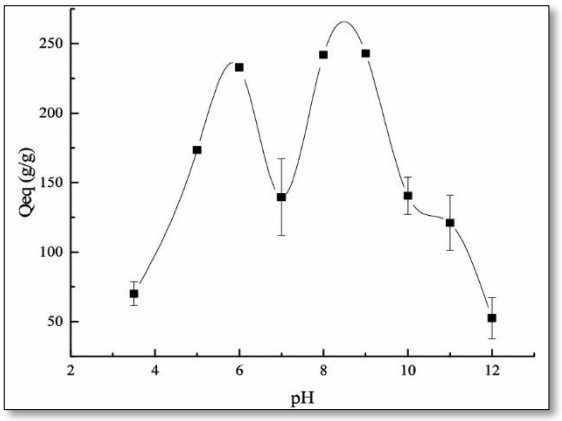


Figure 4.10 Effect of pH on water absorbance

4.4.2.7 Effect of reaction time on water absorbance

As the reaction time grew, the water absorbance first rose and subsequently fell. Early on, there were more crosslinking events and the development of a more sophisticated network structure in the polymer as a result of the polymerization and crosslinking reactions going smoothly. Consequently, the soluble fraction of the polymer dropped and the monomer conversion rate rose, which led to an increase in water absorbance with longer reaction times.

However, many branched chains developed inside the network structure when the reaction time got too long. The polymer's ability to stretch and expand was limited by the entanglement of these chains. Water absorbance decreased as a result of the network of the polymer being harder for water molecules to get through. High absorbance was seen after one hour. 325 g/g was seen highest absorbance at 1 hour.

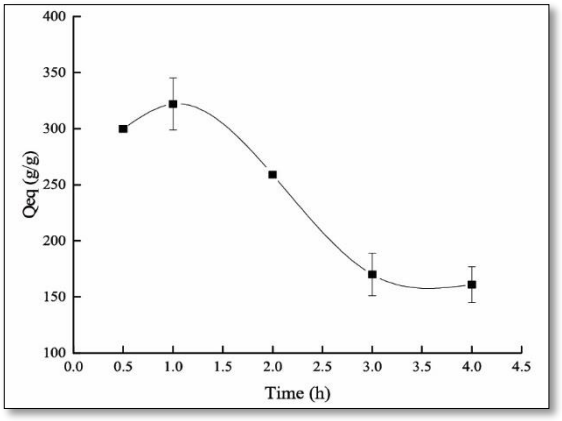


Figure 4.11 Effect of reaction time on water absorbance

CHAPTER 5

DISCUSSION

A breakthrough ecologically friendly and sustainable absorbent material is superabsorbent polymer (SAP) made from pearl millet starch. The starch is then extracted from the millet grains using the process by a sodium hydroxide solution. Instead, this process produces a starch markedly different from that from traditional source having special properties. Superabsorbent polymers are well made from millet starch because of the intrinsic properties of millet starch. Commercially, due to their high swelling capacity and application range, the high use of millet starch would transform to a super absorbent polymer for storing and release water. It was stable, biocompattible and can make up environmental problems associated with applying the conventional non-biodegradable superabsorbent polymers. Although the product has been drying for a long time, it still has a lot of potential as a novel, biodegradable PMSSAP. PMSSAP was evaluated by different techniques like FTIR, SEM, TGA, and XRD, whereas some other quality tests were done to determine the swelling, absorbance, saponification, and biodegradation. The present study confirmed that a strong and wide absorption band of FTIR at 3260 cm^{-1} served as an example of how hydrogen bonds incorporating -OH groups stretch and vibrate. An absorption signal at 2932 cm^{-1} that was only moderately strong suggested that aliphatic structures have both symmetric and asymmetric C-H stretching vibrations. The C-H bonds in methyl groups' bending vibration are thought to be responsible for the peak at 1402 cm^{-1}. The absorption bands at 1552 and 1052 cm^{-1}, respectively, clearly showed that there are -C=O bonds present, which are connected to conjugated ketones, carboxylic acids, and esters. The chains with acrylic acid grafts have a new band, which is indicative of C=O stretching. The occurrence of multiple peaks at similar locations in the FTIR spectra of the grafted starch gels is noteworthy and suggests that MBA, acrylic acid, and starch moieties have been incorporated into the gels' structures. Furthermore, the gels' FTIR spectra showed no signals that may be interpreted as belonging to C=C groups, supporting the lack of free-form acrylic acid in the substance and the total graft copolymerization of all monomers. (29) According to the present study, PMSSAP has

a better, more consistent, and more porous structure, which improves its capacity to absorb water. During the PMSSAP synthesis process, water evaporation caused the creation of these holes. This implied that the space within the cross-linked gel network was expanded by enhanced electrostatic repulsion caused by the carboxylate anion (COO^-) in PMSSAP. In superabsorbent applications, MSSAP has a large specific surface area that promotes quick mass penetration. Additionally, PMSSAP has a compact network structure with significant porosity, which sped up the penetration and retention of water molecules. (28) The porous microstructure increased capillary action and surface area. Water evaporation within the PMSSAP network is the cause of the pore development. The MSSAP has a loose network and little cross-linking, which caused big holes to form as water evaporated. (40) To evaluate the heat stability and breakdown actions of the pearl millet starch-based SAP, thermogravimetric analysis (TGA) was used. The present study revealed that untreated starch lost 10% of its weight at 133 °C as a result of moisture evaporation. Up to 300°C, it maintained remarkable thermal resistance while losing 20% of its weight. Beyond this point, weight loss became apparent and peaked at 50% in the 300–335°C range, which denoted the breakdown of starch molecules. The rate of weight loss decreased as the temperature rose, peaking at a loss of 76.1% at 600°C. This could be linked to the breakdown of amylose and amylopectin, which included steps like saccharide ring dehydration, chain scission that eliminated CO and CO_2, and starch structural fragmentation. (41) The first step happened because of the release of absorbed water with a mass loss, whereas in the second step, the starch backbone degraded and the carbonaceous compounds were removed with a mass loss and a residual mass. (32) It is important to draw attention to a phenomenon where the modified starch decomposes at a lower temperature than the natural starch. This was due to the starch granules' crystalline structure being disturbed during the gelatinization and modification process. (42) The present study showed that the material's improved structural stability is a result of its more refined granular structure. Superior structural stability was also shown by an increased number of peaks in the XRD pattern. The modified starch and millet starch samples' spectra displayed distinct features. Strong diffraction peaks at 15.61, 17.83, 18.14, and 23.79° (2θ) were seen in millet starch in its natural state, when its relative crystallinity was 21.01%, according to patterns found in grain starches.

The observed variations in crystallinity were caused by variations in the size of crystallites and the orientation of double helices inside the starch's crystalline area because the addition of grafted starch caused a noticeable modification in the starch's intensity peak, making it into a wider peak with lower crystallinity as compared to native starch. This showed that the starch structure has undergone a partial transition from a crystalline to an amorphous state. (43) This decrement in crystallinity pointed to a concurrent rise in the amorphous portion of the starch relative to amylopectin. (26) According to the present study, at 6g of millet starch, the absorbance value with the highest value—320.47 g/g—was found. However, at 80 g/g, 8g of starch got the lowest absorbance rating because too much polysaccharide could become too viscous, which prevented graft polymerization from proceeding efficiently and resulted in too many extra cross-link sites in the polymeric structure. This excess of cross-link points raised the cross-link density, which in turn decreased the amount of space in the available polymer network. As a result, the resulting PMSSAP has less water absorbance capability due to this decrease in inaccessible space inside the polymer network. (44) In polymerization procedures, initiators are employed to start and spread the polymerization reaction. A key role in the creation of these polymer chains was played by initiators. Initiators are used in minute quantities. As the present study checked, the water absorbance rose as the KPS concentration increased. However, as KPS concentration climbed further, water absorbance dropped from 325 $g \cdot g^{-1}$ to 240 $g \cdot g^{-1}$. This happened because the KPS initiator created a lot of free-radical reactive sites, which caused many expanding polymer chains to develop quickly before equilibrium was reached. This increased the water absorbance of superabsorbent polymers by establishing a three-dimensional structure. On the other hand, an overabundance of KPS caused an overproduction of free radicals. These extra-free radicals caused a higher percentage of homopolymerization, which produced materials that were soluble in water, as well as the termination of expanding molecular chains, which raised cross-linked density. Consequently, a PMSSAP with reduced polymer network gaps is formed, leading to a reduction in water absorbance. Moreover, the influence of polysaccharide type on the amount of initiator required is similar to the effect of polysaccharide content. (44) The present study confirmed that the absorbance increased from 345 $g \cdot g^{-1}$ when the MBA content was raised from 0.05 weight percent; however, absorbance decreased as the MBA concentration was raised even more.

The network structure became denser with an excess of cross-linkers, which reduced its capacity to absorb water (50 g/g). Owing to an increasing cross-linker concentration caused the production of more expanding polymer chains, which were caused by an increased number of cross-link sites, creating a second network. As a result, the water absorbance decreased as the cross-linker concentration increased. This was due to the superabsorbent polymer system having less free space available, which restricted the number of water molecules that entered its network structure. (45) The network of the PMSSAP could not be developed when the cross-linker content was too low, mainly because there were not enough cross-linking spots. In light of this, PMSSAP has limited water absorption capacity and is only partially soluble. On the other hand, an overabundance of crosslinkers could result in a high cross-link density and a surplus of cross-linking points, which could limit the network's growth and decrease the amount of area that water could enter. As such, the superabsorbent polymers demonstrated a reduced ability to swell. (44) According to the present study, enhancing the ionic strength inside the network is a useful method for enhancing salt tolerance. Salt samples like NaCl and KCl solutions at varied concentrations demonstrated how different cations affected the superabsorbent polymer's ability to absorb. When the PMSSAP was immersed in electrolyte solutions, the network contracted, which resulted in a constant drop in the swelling capacity as the concentration of the salt solution increased. The screening action of positive cation charges is thought to be the cause of the reduction in the extent of swelling of superabsorbent in salt solutions in contrast to absorption in undiluted water. Osmotic pressure between the internal solution of the hydrogel along the exterior solution decreased as a result of anions in the system repelling one another electrostatically. In salt solutions containing polyvalent cations as opposed to those with univalent cations, PMSSAP swelling was significantly decreased. Multivalent cations could also cause the polymer matrix to undergo a process known as ionic cross-linking. As a result, when cation valence increased, the absorption of the gel significantly declined. (29) It was therefore obvious that absorbance rose as the temperature rose. The resultant PMSSAP has a relatively reduced molecular weight and water absorption capacity due to the lessened radical activity, slowed initiator breakdown rate, and hampered chain propagation at lower temperatures. On the other hand, higher reaction temperatures promoted monomer conversion, increasing the capacity for water absorption. (33) pH also

determines the extent of absorbance. According to the present study, the superabsorbent polymer's absorbance capability was assessed in solutions with pH ranges of 4 to 12.0. The PMSSAP's absorbance increased as the pH climbed from 4 to 6.0, then fell to pH 7.0. It then climbed once again to pH 8.0, after which it kept decreasing. The acrylic acid groups in the polymer network changed at high pH values, which attracted cations to replace H^+ ions in the polymer region. As a result, the PMSSAP's free ion concentration increased, increasing the ionic swelling pressure. As a result, PMSSAP grew, increasing the repulsion between the groups of ionized polyacrylates. At pH 8, when all of the acrylic acid groups had changed, the MSSAP's water absorbance was at its maximum. The PMSSAP 's higher swelling capacity was caused by an increased anionic density. The combination of enhanced ionic strength and the shielding effect (screening effect) produced by the high concentration of Na^+ cations, which come from NaOH, could be blamed for the decrease in swelling above pH 8. (46)

CONCLUSIONS

The development of Pearl Millet Starch-based Superabsorbent Polymer (PMSSAP) represents a significant advancement in sustainable water-absorbing materials because it shows how natural and synthetic elements can create high water-retaining superabsorbent polymers (SAPs) effectively. The synthesis of PMSSAP involved polymerization between acrylic acid (AA) and acrylamide (AM) under cross-linking mechanisms from methylene bis-acrylamide (MBA) alongside potassium persulfate (KPS) initiation and assuming millet starch as the natural polymeric foundation. TGA combined with FTIR along with SEM and XRD analyses provided data about the properties of PMSSAP. The TGA analysis determined the thermal stability of PMSSAP which demonstrated suitable usage in heat-resistant applications since it maintained structural properties during elevated temperatures. SEM imaging revealed that PMSSAP's surface has a porous shape which creates better water intake. Efficient superabsorbent polymers function effectively through rapid water uptake combined with retention capability because of the linking pores structure. A range of conditions can be assessed using XRD analysis to verify the PMSSAP structure remains stable. Water absorption reached its best point at 0.05% methylene bis-acrylamide concentration because the polymer could absorb 345 g/g of water. A proper concentration of cross-linker leads to maximum water retention. The water absorption level of PMSSAP fell heavily after it came into contact with salt solutions. These typical superabsorbent phenomena emerge from the competition between salt ions and polymer network which reduces swelling properties. The cross-linked PMSSAP polymer demonstrated both strong thermal stability and effective absorption properties which make it suitable for various applications.

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