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Research Article

Optimization of Cassava-Peel Derived Nanostarch Via Sulphuric Acid Hydrolysis Using Taguchi Method

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Abstract

Untreated cassava peel waste generated during harvesting and processing poses significant environmental challenges. Synthesis of starch nanoparticles from cassava peels for various applications offers a sustainable solution to waste reduction and contributes to environmental conservation. The unique characteristics of nanostarch such as thermal stability, high solubility, non-toxicity, and low cost enable its application in the food industry, cosmetics, enhanced oil recovery, and textiles. The current study employed the Taguchi method design to optimize sulphuric acid hydrolysis in synthesizing cassava peel-derived nanostarch. Additionally, the derived cassava peel nanostarch was characterized using Fourier Transform Infrared Spectroscopy (FTIR). Starch was extracted from cassava peels, followed by synthesizing starch nanoparticles via sulphuric acid hydrolysis. Optimization of nanostarch synthesis was based on randomized experimental runs using the Taguchi method generated by the Minitab software, with the experiments conducted in duplicates. The optimum conditions for the experiment were found to be 3 hours, at 25 ℃ using an H_2SO_4 acid concentration of 2M. These conditions produced a yield of 92.28%. ANOVA analysis identified sulphuric acid concentration as the most significant factor that affected cassava nanostarch yield, with p-values of 0.026 and 0.003 for the signal to noise (S/N) ratios and means, respectively. The least significant factor based on the analysis was the hydrolysis time. However, according to the S/N ratios main effect plot, the most optimum conditions predicted by the Taguchi method design was 9 hours, 25 ℃ using H_2SO_4 acid concentration of 2M. A confirmation experiment conducted at 25 ℃, using an H_2SO_4 acid concentration of 2M for 9 hours gave a nanostarch yield of 97.01%. In conclusion, the Taguchi method design identified sulphuric acid concentration as the most significant factor in synthesizing cassava peel-derived nanostarch via acid hydrolysis.

Keywords

Cassava-Peel Nanostarch, Taguchi Method, Optimization, Sulphuric Acid Hydrolysis

1. Introduction

Cassava (*Manihot esculenta*) thrives in lowland tropical regions, performing best in warm, moist climates with temperatures ranging from 19-25 ℃ and annual rainfall of 100-150 cm [1]. Cassava, one of the major sources of starch in sub-tropical and tropical areas, can be used as a raw material for the creation of biodegradable polymers [1]. Starch obtained from cassava is different from other sources of starch due to its reduced low residual material content, amylose

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content, and high molecular weight amylopectin and amylose [1]. Cassava by-products, including as leaves, peels, stems, and seivate, can be utilised as feedstock or as substitute substrates for biotechnological processes as a helpful strategy to mitigate environmental issues [2].

The cassava peels, which constitute about 15% of the cassava root's total weight [3], are agricultural waste commonly generated during cassava tuber peeling. It is a waste usually dumped indiscriminately and constitutes environmental pollution, which endangers terrestrial life. These peels easily rot after some days of disposal and produce foul odors polluting the environment due to microbial activities such as fungi, yeast, and bacteria. This waste has been disposed-off without adequate control measures and this has led to the emittance of obnoxious smell and posing health risks [4]. Additionally, vegetation and soil in areas designated for dumping cassava peels become unproductive and devastated due to biological and/or chemical reactions taking place as the peels are degrading [5].

Starch exists in abundance as a natural, renewable biodegradable polymer that is produced by many plants as a source of energy. This polymer is made up of amylose and amylopectin molecules. Amylose is a linear polysaccharide, and amylopectin is a branched chain polymer [6]. Starches are not suitable for most uses due to their low shear and thermal stability as well as a high retrogradation rate. Additionally, native starches are prone to syneresis aside from the gelling tendency of the pastes; thus, require alteration to enhance their desirable characteristics and/or minimize their defects [7]. According to Alves et al. [8], modification of starch macromolecules from micro to nanoscale not only alters the particle size but also has an effect on their functional properties. There are two types of nanostarches: starch nanoparticles (SNPs) made from gelatinised starch, which may contain amorphous areas, and starch nanocrystals (SNCs), which are crystalline sections created from the breakdown of the amorphous phases inside the starch granules [9].

Researchers modify starch using physical, chemical, and enzymatic methods, or combinations, to improve its properties for application in various industries [10]. Physical methods modify starch molecules by thermal and non-thermal techniques [11]. Chemical modification techniques involve adding new chemical groups to starch molecules, which enhances the starch's functional properties [12, 13]. These properties include less retrogradation, increased viscosity, decreased gelatinisation temperature, and increased processing stability. By creating more resistant starch through enzymatically catalysed debranching of the starch molecules, enzymatic modification modifies the functionality of starch [12]. Since acid hydrolysis yields starch nanoparticles that are more stable and have a considerably higher crystallinity than other procedures, it is the recommended approach for preparing nanostarch [14].

A variety of variables influences the process of acid hydrolysis, which ultimately results in the development of nanostarch with distinct mechanical, chemical, and technical characteristics [15]. Three key parameters that have a major impact on the process are reaction time, temperature, and acid concentration [15]. The origin and crystallinity of starches, the type and strength of acid, the time and reaction temperature affect the degree of hydrolysis and, as a result, have a significant effect on the yield and quality of acid hydrolysed SNCs [16]. According to Qin et al. [17], current acid hydrolysis procedures have limited practical application due to their low ultimate yield, time-consuming nature, and high cost. As a result, it is critical to develop an alternative method that enhances the process by increasing yield while reducing reaction time.

Low nanoparticle yield leads to greater production costs, hence increasing yield leads to considerable cost savings. Process optimization is the most effective way to increase production yield [15]. The Taguchi approach in experimental design identifies the optimal parameter values to reduce production quality variance. This technique also reveals the most important components that lead to variance [18]. By using the Taguchi method approach, the parameters that offer the best performance and efficiency can be identified thus saving time, cost, and resources by eliminating the need for repeated trials [19]. The signal-to-noise (S/N) ratio is computed using the loss function's value, which is also used to determine performance metrics that deviate from the intended goal value. Three categories are often used to group performance: "smaller the better," "nominal the better," and "higher the better" statistics [20]. According to Pardo [21], this approach uses fractional factorial experiment design to decrease the number of trials, resulting in cost savings and time efficiencies. Following the laboratory experiments, the obtained data is converted into the Signal-to-Noise (S/N) ratios using the quality parameters specified. The bigger the S/N ratio number, the better the response of interest quality. Analysis of Variance (ANOVA) identifies the most relevant process factors that influence statistical quality characteristics [18].

In the current study, the synthesis of starch nanoparticles from cassava peels was explored as a solution to remediate the environmental challenges posed by cassava waste, with the Taguchi method employed to optimize the sulphuric acid hydrolysis process, resulting in a significant improvement in nanostarch yield and its applicability across multiple industries.

2. Materials and Methods

2.1. Chemicals and Materials Used

Cassava tubers were collected from the farm; and peeled to obtain the cassava peels that were used to extract starch. Sulphuric acid was obtained from Science Lab Kenya Limited. Distilled water and apparatus (magnetic stirrers, beakers, and conical flasks) were obtained from the laboratory at the Technical University of Kenya.

2.1.1. Extraction of Starch from Cassava Peels

The cassava peels were washed to remove the brown part and any other debris and were blended into a pulp. The pulp was mixed with water and filtered using a muslin cloth. The resulting liquid was left to stand overnight. The filtrate was decanted to obtain the white cassava starch that had settled at the bottom. The white paste was left to sun dry to give a powdery cassava starch.

2.1.2. Optimization of Sulphuric Acid Hydrolysis of Cassava Peel Starch Using Taguchi Method

The experiments were designed based on orthogonal arrays to optimize the Sulphuric acid hydrolysis of cassava peel starch for cassava peel nanostarch production. Independent variables, time (A), temperature (B), and acid concentration (C) were optimized in this experimental design for optimal nanostarch yield (response of interest). The experiment was conducted at nine experimental points generated by the Minitab Software. The levels of each independent variable and the experimental design matrix are presented in Table 1. The experimental factor levels were chosen based on literature values used for cassava nanostarch production [18].

According to Sabarish et al. [22], three standard S/N equations are widely used to categorize the objective function - larger the better, smaller the better, or nominal the best. A larger is better was chosen since the study's aim was to maximize the response of interest. Equation 1 shows how the S/N is calculated for the "larger the better" function;

$$\frac{S}{N} = -10 * log\ log \left(\frac{\Sigma\left(\frac{1}{Y2}\right)}{n} \right) \quad (1)$$

Where Y is the response for the given parameter level combination and n is the number of responses in the factor level combination.

The experiments were conducted in a randomized order, generated by the Minitab software. An L_9 orthogonal array was selected for this study as shown in Table 1.

2.1.3. Batch Sulphuric Acid Hydrolysis Studies for Nano Starch Production

The cassava peel sulphuric acid hydrolysis experimental runs were conducted following the designed Taguchi experimental model. The experimental procedure involved dispersing 15 g of cassava peel starch in diluted sulphuric acid solution according to the experimental design matrix in 250ml conical flasks and the dispersion was magnetically stirred at 100 rpm. After various durations of acid hydrolysis, the suspensions were filtered with successive washing. The percentage yield was calculated as shown in Equation 2 [23]:

$$Recovery\ yield\ (\%) = \left(\frac{Wa}{W}\right) x\ 100\% \quad (2)$$

Where: W_a is the weight of starch (dry basis) after acid hydrolysis, and W is the weight of starch (dry basis) before acid hydrolysis.

2.2. Characterization of Cassava Peel Starch

Cassava peel starch chemical structure was examined using Fourier Transform Infrared Spectroscopy (Thermo Scientific, TruDefender FX6455) before and after sulphuric acid hydrolysis. The samples were dried at 105 ℃ for 2 hours before analysis to avoid moisture interference.

3. Results and Discussion

3.1. Optimization Using Taguchi Method

Table 1 shows the nanostarch yield percentages in each of the experimental runs conducted according to the Taguchi method design. Each test combination was replicated two times. It can be seen that the optimum conditions for cassava peel nanostarch production were within 3 hours at 25 ℃ at an acid concentration of 2M.

According to the Taguchi method design, once the cassava peel nanostarch yields are obtained, the data is converted to a measure of variability known as the signal-to-noise ratio (S/N). Based on this analysis, the larger the better performance characteristic was chosen according to Nguyen et al. [24] because the study's aim was to maximize the yield of the nanostarch. Nanostarch synthesized with a higher acid concentration gave a lower percentage yield. This is due to corrosion and degradation at high acid concentrations, which causes more cleavage of the starch glyosidic linkages and results in a reduction in recovery yield. Saeng-on et al. [25] discovered a similar relationship between acid concentration and nanostarch yield. They reported that nanostarch synthesized at higher acid concentration (4.5M H_2SO_4) resulted in a lower percentage yield. Chen et al. [26] conducted a study on the yield pattern of nanocellulose from bleached eucalyptus kraft dry lap pulp by varying H_2SO_4 concentrations from 50 to 64-wt%. The results suggested that the nanocellulose yield reduced rapidly as the H2SO4 concentration above 58-wt%.

***Table 1**. Taguchi optimization experimental design (L_9).*

Time (hours)	Temperature (℃)	Acid Concentration (M)	%Experimental yield	S/N ratio
3	25	2	92.28	39.30215
3	40	3.5	74.36	37.42679
3	55	5	7.04	16.95145
6	25	3.5	85.23	38.61185
6	40	5	2.96	9.425834
6	55	2	85.45	38.63424
9	25	5	11.79	21.43028
9	40	2	85.14	38.60267
9	55	3.5	67.22	36.54997

3.2. Analysis of Variance (ANOVA)

Analysis of Variance (ANOVA) method is a well-established method used to determine the percentage contribution of each process parameter on the desired outputs [27]. ANOVA model was performed to find the significance of factors such as sulphuric acid concentration, time, and temperature on the yield of nanostarch in different working conditions. The ANOVA tables provide valuable insights into the relative importance of each factor. The p-values of Tables 2 and 3 indicate which variables had a significant impact on the interested response. By examining the p-values, the level of significance for each parameter can be determined and prioritized. According to Abebe et al. [28], if the factors' p-value is less than 0.05, it is presumed that the factor has a significant influence on the output response, but if 0.05 is less than the *p*-value, the factor does not affect the output response.

Tables 2 and 3 show the results of ANOVA with the S/N ratios and means, respectively, as per the Taguchi experimental design. According to the ANOVA analysis, acid concentration was the most significant factor with a p-value of < 0.05. From Table 2, it can be observed that in the case of the S/N ratios, sulphuric acid concentration was the most significant factor with a p-value of 0.026 and the hydrolysis time was the least significant factor with a p-value of 0.606.

Table 3 shows that in the case of the means, concentration was the most significant factor with a p-value of 0.003 and the hydrolysis time was the least significant factor with a p-value of 0.653.

***Table 2**. Analysis of Variance for SN Ratios (Experimental Data).*

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Time (hours)	2	17.31	17.31	8.654	0.65	0.606
Temperature (℃)	2	32.17	32.17	16.083	1.21	0.452
Acid Concentration (M)	2	992.91	992.91	496.456	37.36	0.026
Residual Error	2	26.58	26.58	13.290		
Total	8	1068.97				

***Table 3**. Analysis of Variance for Means (Experimental Data).*

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Time (hours)	2	20.1	20.1	10.05	0.53	0.653
Temperature (℃)	2	178.2	178.2	89.08	4.71	0.175

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Acid Concentration (M)	2	11272.6	11272.6	5636.28	297.69	0.003
Residual Error	2	37.9	37.9	18.93		
Total	8	11508.7				

The order of the process parameters is determined based on the S/N ratio values shown in Table 4. This ranking is decided by comparing delta values, which represent the difference between maximum and minimum values for levels of each factor [21]. A design factor that significantly varies the signal-to-noise ratio from one factor setting to the next suggests that it significantly contributes to attaining the performance characteristic [10]. A factor may not have much of an impact on the performance characteristic if the signal-to-noise ratio hardly changes across factor settings. Based on the results, the acid concentration had the most significant effect on the yield of nanostarch.

Table 4. *Response Table for Signal-to-Noise Ratios (Experimental Data).*

Larger is better

Level	Time (hours	Temperature (℃)	Acid Concentration (M)
1	31.23	33.11	38.85
2	28.89	28.49	37.53
3	32.19	30.71	15.94
Delta	3.30	4.63	22.91
Rank	3	2	1

Table 5, the response table for the means further reinforces the significance of sulphuric acid concentration as the primary driver of the cassava peel nanostarch yield.

Table 5. *Response Table for Means (Experimental Data).*

Level	Time (hours/	Temperature (℃)	Acid Concentration (M)
1	57.893	63.100	87.623
2	57.880	54.153	75.603
3	54.717	53.237	7.263
Delta	3.177	9.863	80.360
Rank	3	2	1

To visualize the variation of response with the change in the levels of a parameter, the response curves were used. The mean of means represents yields that are calculated based on the yields obtained at each level of each factor. From the graph, it can be noted that the most significant conditions were at 25 ℃, within 3 hours, and at 2M acid concentration as shown in Figure 1.

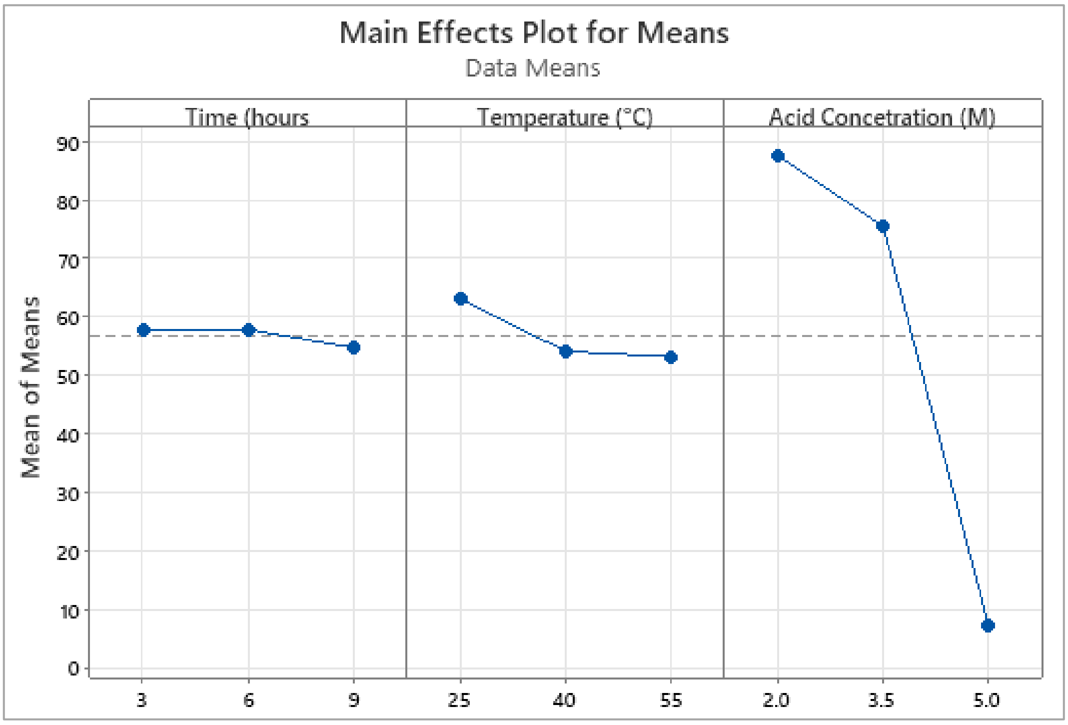

Figure 1. *Main effects plot for means.*

The main effect plot for S/N ratios in Figure 2 clearly shows the optimal settings for each factor, with acid concentration being the most significant. It is observed from the main effect plot using the S/N ratio of yield values that the optimal manufacturing parameters are acid concentration: 2.0 M, hydrolysis temperature: 25 ℃, and hydrolysis time: 9 hours as shown in a confirmation test that was conducted and the conditions produced an average nanostarch yield of 97.01%.

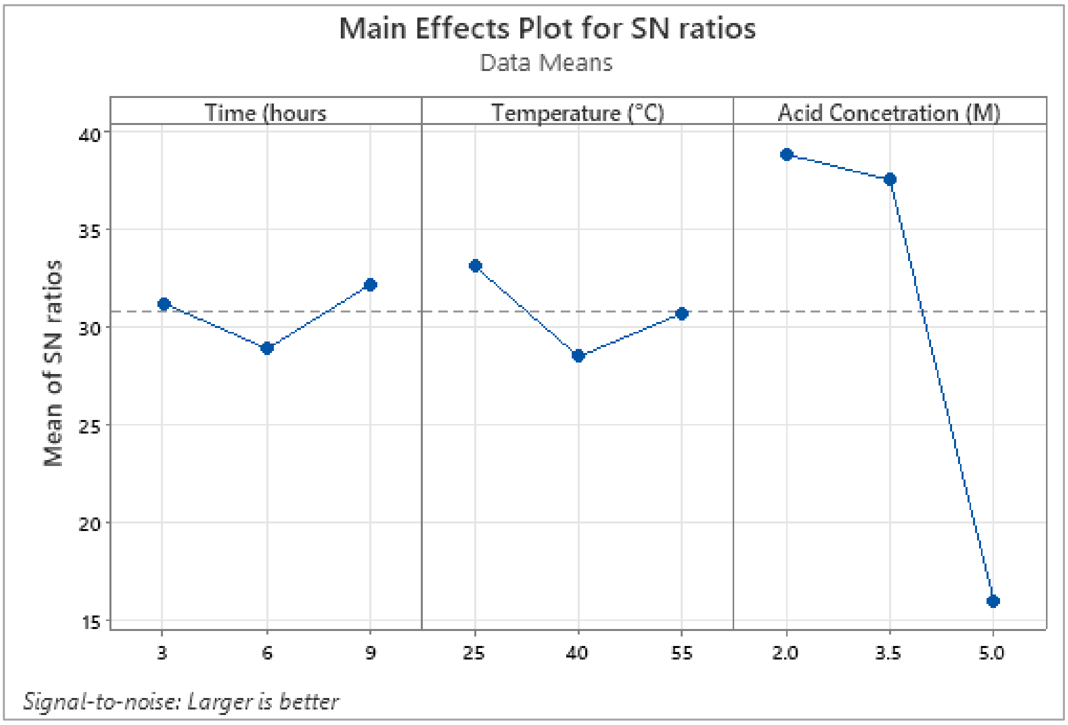

Figure 2. *Main effects plot for SN ratios.*

3.3. Characterization of Cassava-Peel Starch

The FTIR spectra of cassava peel starch before and after sulphuric acid hydrolysis are shown in Figure 3. Acid-hydrolyzed cassava peel starch exhibited the same distinctive peaks as cassava peel native starch. A prominent, broad peak at approximately 3260 cm^{-1} corresponds to O-H stretching vibrations of hydroxyl groups in glucose monomers. The distinctive peak at 2929 cm^{-1} corresponds to C-H stretches [29]. The peaks at 1150 cm^{-1} and 1080 cm^{-1} suggested the stretch of C-O-C links in glucosidic rings, whereas the absorption peak at 1641 cm^{-1} revealed the H-O-H bending vibrations in the starch-bound water [25].

Additionally, the absorbance peak at 1420 cm^{-1} and 1341 cm^{-1} indicates the C – H bend of CH_2OH moiety. A sharp absorption peak at 998 cm^{-1} represents a C – O stretch. Absorption peaks at 930 cm^{-1}, 855 cm^{-1} and 760 cm^{-1} correspond to the C – O – C ring vibration of carbohydrates [29]. According to the spectra, starch's chemical structure was similar before and after sulphuric acid hydrolysis, but the absorption peaks position and intensities only slightly changed, reflecting the modification of the starch molecular groups and the short-range order degree of the starch particles [30].

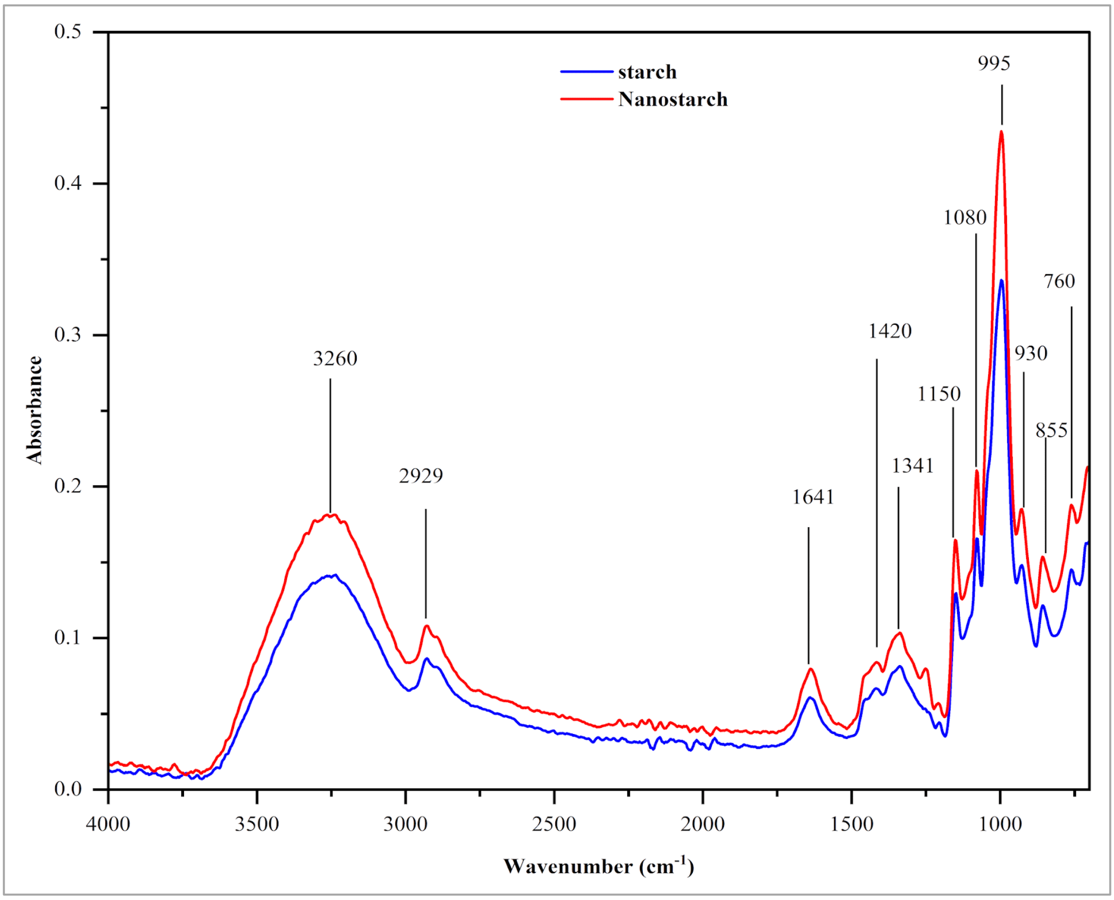

Figure 3. FTIR spectra for cassava peel starch before and after acid hydrolysis.

4. Conclusion

Taguchi method design was found to be effective in the optimization of cassava peel nanostarch yield with the design showing that the optimum yield was at 25 ℃, for 3h at an acid concentration of 2M. Based on the main effect plot for S/N ratios, the optimum conditions were found to be 25 ℃, for 9 hours at an acid concentration of 2M. In conclusion, time, temperature, and acid concentration have a statistically significant effect on the yield of the nanostarch produced via sulphuric acid hydrolysis with acid concentration as the most significant factor as illustrated by the main effect plots for S/N ratios and means. Overall, the findings demonstrate the potential of cassava peels as a source for production of nanostarch for various industrial applications. In order to improve its usefulness in certain applications, such drug delivery or food preservation, future studies may concentrate on modifying cassava peel nanostarch surface properties. Its application in many industrial sectors would also be optimized with a thorough assessment of its characteristics, such as crystallinity, particle size, and thermal stability.

Abbreviations

SNCs	Starch Nanocrystals
SNPs	Starch Nanoparticles
ANOVA	Analysis of Variance
S/N	Signal to Noise Ratio

Author Contributions

Jael Kanyiri: Conceptualization, Resources, Writing – original draft, Data curation

Frank Ouru Omwoyo: Methodology, Software, Writing – reviewing and editing, Validation, Formal analysis

Patrick Musyoki Shem: Writing – reviewing and editing, Validation, Funding acquisition

Geoffrey Otieno: Supervision, Project Administration, Writing – reviewing and editing, Funding acquisition

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Data Availability Statement

The data is available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare no conflicts of interest.

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Biography

Jael Kanyiri is currently a student at the Technical University of Kenya (TU-K) pursuing a Bachelor’s degree in Industrial Chemistry having graduated from the same institution with a Diploma in Analytical Chemistry in 2019, Her expertise includes wet Chemistry and synthesis of nanomaterials with a focus in sustainable waste management. Her expertise highlights her commitment to advancing environmental solutions through innovative chemical processes.

Frank Ouru Omwoyo is a Research Assistant to Dr. Geoffrey Otieno at the Technical University of Kenya (TU-K). He graduated with a bachelor’s degree in Industrial Chemistry from TU-K in 2023. His expertise includes designing experiments, data modeling, and analysis using advanced statistical software. With a strong foundation in chemistry, he is keen to advance his academic journey by pursuing further studies and conducting research in the fields of inorganic and physical chemistry, where he hopes to contribute to innovative solutions in these areas.

Geoffrey Otieno is currently the Director of The School of Chemistry and Material Science at The Technical University of Kenya. Geoffrey obtained a PhD in Material Science from the University of Oxford, UK in 2012; and a Master's in Advanced Materials Engineering from Kangwon National University, South Korea (2007). He is a member of the Kenya National Academy of Sciences. He has been a member of the Governing Council of Kenya Chemical Society (KCS) since 2015. He is a fellow of the Organization of the Prohibition of Chemical Weapons (OPCW) having completed the Associate Program in 2017. Geoffrey's career as a professional Chemist and Material Scientist spans over 20 years. These years have been spent as a researcher, and advisor to government, international organizations, and private organizations. As an academic, Dr Otieno worked at Kangwon National University, South Korea as a researcher on the development of bipolar plate materials for fuel cells supported by Samsung and as a post-doctoral researcher at Oxford Materials on novel nanostructures for applications in electronics and body armor. His research interests are in the area of nanomaterials with applications in water treatment and renewable energy. Recent key projects include leading research with Nestle Waters (France) in formulating biodegradable polymers and the University of Oxford on Solar concentrators for bone charring.

Patrick Musyoki Shem is a Senior Lecturer of Chemistry in the School of Chemistry and Material Science at The Technical University of Kenya. He graduated with a PhD in Chemistry from the University of Utah, Salt Lake City in 2012. He is a holder of BSc (Hons) and MSc degrees in chemistry from the University of Nairobi. From 2012-2013, he was a Postdoctoral Research Associate at the Material Science Institute, University of Oregon in Eugene where he worked in the Safer Nanomaterials and Nanomanufacturing Initiative. His expertise includes the synthesis of nanomaterials and, the application of spectroscopic, surface analytical, and microscopy techniques in the characterization of nanomaterials. He is also an expert in chemical safety, security, and chemical management.

Research Field

Jael Kanyiri: Synthesis of Nanomaterials, wet chemistry, environmental pollution

Frank Ouru Omwoyo: Catalysis, functional materials, wastewater treatment, energy conversion and storage, surface chemistry

Geoffrey Otieno: Nanomaterials with applications in water treatment and renewable energy

Patrick Musyoki Shem: Synthesis and application of new materials for drug delivery, diagnostics, sensors, catalysis, environmental analysis and remediation; toxicology of nanomaterials