

## EFFECTS OF SOAKING PRETREATMENT AND CITRIC ACID LINTNERIZATION ON THE PHYSICOCHEMICAL AND FUNCTIONAL PROPERTIES OF RED GUINEA CORN STARCH

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### ABSTRACT

This study investigated the influence of pretreatment and citric acid lintnerization on the physicochemical and functional properties of Red Guinea corn starch. Cereal grains were soaked at room temperature for varied durations (0 h, 24 h and 48 h) before starch isolation, after which the starch was subjected to citric acid lintnerization at different concentrations (1 M, 3 M and 5 M). The effects of soaking time were evident in the subsequent esterification process, with prolonged soaking enhancing solubility (% S) and Paste clarity (%PC) while reducing swelling power (SP) and water holding capacity (WHC). SP decreased from  $10.89 \pm 1.13$  g/g in unsoaked crude starch (U-Cs) to  $5.33 \pm 0.35$  g/g in 5 M lintnerized starch subjected to 48 hours soaking (S<sub>48</sub>-M<sub>5</sub>). Solubility increased with increase in citric acid concentration, reaching  $32.00 \pm 11.1\%$  in S<sub>48</sub>-M<sub>5</sub> compared to  $8.00 \pm 0.0\%$  in unsoaked crude starch (U-Cs). Similarly, paste clarity increased with prolonged lintnerization, from  $23 \pm 0.00\%$  in U-CS to  $59 \pm 0.00\%$  in S<sub>48</sub>-M<sub>5</sub>. FTIR analysis confirmed the presence of a new peak at  $1718 \text{ cm}^{-1}$ , which is attributed to the presence of C=O carbonyl stretches of ester. Also, there is a sharp peak at  $1541 \text{ cm}^{-1}$  which is attributed to C-O-C stretch of ester. Which were absent in the spectra structure of the crude starches. The degree of substitution increases (from  $0.5427 \pm 0.00$  to  $0.7817 \pm 0.00$ ) with increase in citric acid concentration. These findings demonstrate that soaking pretreatment alters starch granule accessibility and reactivity, thereby intensifying the effects of citric acid lintnerization.

**Keywords** Pretreatment, Lintnerization, Citric Acid, Red Guinea Corn

### INTRODUCTION

Starch is a fundamental biopolymer widely employed in food and pharmaceutical industries due to its thickening, gelling, and stabilizing properties (Hughes, et al., 2020). However, crude starches often display limitations such as restricted solubility, poor pasting properties, and limited functional diversity, which necessitate modification to enhance their performance (Berski et al., 2011). Chemical modifications, particularly acid hydrolysis and esterification (lintnerization), have been employed to improve starch properties by altering granule structure and molecular composition.

Pretreatment of starch prior to modification has emerged as a critical step in determining the efficiency and outcome of subsequent chemical reactions. Soaking cereal grains in water for extended periods at room temperature can induce partial hydration, leaching of soluble components, and structural rearrangements within the starch granules. These changes may influence the accessibility of hydroxyl groups, the susceptibility of starch chains to hydrolysis, and the extent of esterification during citric acid lintnerization. Thus, soaking duration represents a key variable that can modulate the physicochemical response of starch to acid modification. Citric acid, a safe and food grade organic acid, is particularly attractive for starch modification because it not only hydrolyzes glycosidic linkages but also promotes

esterification, leading to cross-linking and improved functional attributes. When combined with pretreatment strategies such as soaking, citric acid lintnerization may produce starches with enhanced solubility, reduced swelling power, and altered polymer content, thereby expanding their potential applications in food formulations and biodegradable materials.

Red Guinea corn (*Sorghum bicolor*) as is an underutilized cereal that offers a promising alternative source of starch. Sorghum grain contains 68-75% starch depending on the climatic conditions and cultivar (Karmvir et al., 2018). Sorghum is free from gluten which makes it useful for a person suffering from celiac disease. Sorghum starch has been reported to have more functional properties compared to that of corn starch (Karmvir et al., 2018). Yet, limited studies have examined how soaking time prior to esterification influences the physicochemical and functional properties of its starch. This research therefore investigates the effect of soaking time and citric acid lintnerization on red guinea corn starch. The study aims to elucidate how soaking pretreatment conditions shape the modification process, providing insights into optimizing starch functionality for diverse industrial applications. Figure 1 showed red guinea corn samples prior to soaking.

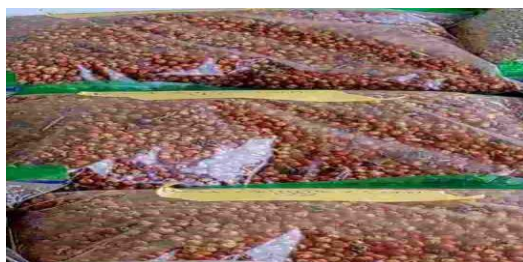


Figure 1: Red Guinea Corn Sample

## MATERIALS AND METHODS

### Extraction of Starch

The extraction of starch from red guinea corn cereal was carried out following the method employed by Raugrussmee and Anal, 2019. Cereal sample (4.5 kg) was divided into three portions; The first portion, sample (U-Cs-RGCS) was washed thoroughly in clean water and ground immediately with water (1:2 w/v) at room temperature, 25°C until a smooth slurry was formed in a blender. The slurry was sieved in a 150 µm pore size sieve. The residual flesh on the sieve was further washed with water twice to remove the remaining starch. The filtrate was allowed to stand for 2 hrs to allow the starch sedimentation. The starch was separated by decantation and dried in an oven at 40°C and allowed to cool. The dried starch cake was reground with an electric grinder in to a smooth powder and kept in air tight container for further analysis (figure 2) (Raugresmme and Anal (2019).

The second portion sample (S<sub>24</sub>-Cs-RGCS) was soaked in water at room temperature (25°C) for 24hrs, then the grains were thoroughly washed using fresh, clean water followed by

grounding with water (1:2 w/v) until a smooth slurry was formed in a blender. The slurry was sieved in a 150 µm pore size sieve. The residual flesh on the sieves was further washed with water twice to remove the remaining starch. The filtrate was allowed to stand for 2 hrs to allow the starch sedimentation. The starch was separated by decantation and dried in an oven at 40°C.

The third portion sample (S<sub>48</sub>-Cs-RGCS) was soaked in water at room temperature (25°C) for 48hrs, then the grains were thoroughly washed using fresh, clean water at room temperature, followed by grounding with water (1:2 w/v) until a smooth slurry is formed in a blender. The slurry was sieved in a 150 µm pore size sieve. The residual flesh on the sieves was further washed with water twice to remove the remaining starch. The filtrate was allowed to stand for 2 hrs to allow the starch sedimentation. The starch was separated by decantation and dried in an oven at 40°C for 48 hrs. The dried starch was stored separately in air tight container for further analysis (Torres *et al.*, 2020).



(a)



(b)

Figure 2: Extract of RGCS

### Lintnerization of Starch Samples (Esterification Method)

The pretreated red guinea corn starch samples was lintnerized by using citric acid 1 M, 3 M and 5 M respectively, coupled with heat treatment method following the method employed by Kalu *et al.*, (2022).

#### Procedure for 1 M Lintnerization

An accurately measured 50 ml of 1 M citric acid solution (19 g of the salt dissolved in 50 ml of water) was weighed into a clean 250 ml beaker, the pH of the solution was adjusted to 3.2 using a pH meter by adding 10% NaOH and make up with water to 100ml. Then 100 g of starch sample was added (1:1 w/v) and stirred thoroughly until a smooth slurry is formed. The mixture was allowed to stand for 18 hrs (figure 3) and the starch suspension was then poured out into a flat tray and dried at 40°C until all the water was almost gone. After that the starch cake was superheated at 90°C for 1hr and then cooled to room temperature. the esterified starch was ground and kept in an air tight container for further analysis (Kalu *et al.*, (2022).

#### Procedure for 3 M Lintnerization

An accurately measured 50 ml of 3 M citric acid solution (29 g of the salt dissolved in 50 ml of water) was weighed into a clean 250 ml beaker, the pH of the solution was adjusted to

3.2 using a pH meter by adding 10% NaOH and make up with water to 100ml. Then 100 g of starch sample was added (1:1 w/v) and stirred thoroughly until a smooth slurry is formed. The mixture was allowed to stand for 18 hrs. the starch suspension was then poured out into a flat tray and dried at 40°C until all the water was almost gone. After that the starch cake was superheated at 90°C for 1hr and then cooled to room temperature. the esterified starch is ground and kept in an air tight container for further analysis (Kalu *et al.*, (2022).

#### Procedure for 5 M Lintnerization

An accurately measured 50 ml of 5 M (48 g of the salt dissolved in 50 ml of water) citric acid solution was weighed into a clean 250 ml beaker, the pH of the solution was adjusted to 3.2 using a pH meter by adding 10% NaOH and make up with water to 100ml. Then 100 g of starch sample was added (1:1 w/v) and stirred thoroughly until a smooth slurry is formed. The mixture was allowed to stand for 18 hrs. the starch suspension was then poured out into a flat tray and dried at 40°C until all the water was almost gone. After that the starch cake was superheated at 90°C for 1hr and then cooled to room temperature. the esterified starch is ground and kept in an air tight container for further analysis (Kalu *et al.*, (2022).



Figure 3: Lintnerization of RGCS

### FTIR Analysis of Crude and Lintnerized Red Guinea Corn Starch

Chemical Composition of the crude and lintnerized red guinea corn starches was analyzed by Fourier Transform Infrared (FTIR) spectrophotometer (Agilent Technology Cary 630). Using potassium bromide pellets. Samples were scanned from 400 to 4000 $\text{cm}^{-1}$  at a resolution of 4  $\text{cm}^{-1}$  and the spectra were recorded for total scans.

### Degree of Substitution

The method described by kalu et al., 2022 was used to determine the DS of the lintnerized starch. 1g of the starch sample was dissolved in 25ml distilled water, 25mls of 0.5M sodium hydroxide added and stirred thoroughly for homogeneity, then the solution was heated in a water bath at 60°C for 30 mins with continuous stirring. The solution was allowed to cool to room temperature. Then, 3 drops of phenolphthalein solution was added and the excess sodium hydroxide was titrated against 0.5 M HCl to colorless endpoint. The same procedure was repeated for the blank. The DS was calculated using the formula

$$DS = \frac{162 M(B-S)}{1000 W} \quad \text{Eqn. 1}$$

Where M is the Molarity of HCl (0.5 M), W is the weight of sample (1g), B is the volume of 0.5 M HCl in the blank (ml) and S the volume of 0.5 M HCl in the sample (ml). 162 stands for the molecular weight of anhydro glucose unit (AGU) of a starch molecule ( $\text{C}_6\text{H}_{10}\text{O}_5 = 162$ )

### Thermogravimetric Analysis of the Crude and Lintnerized Red Guinea Corn Starch

Thermogravimetric analysis (TGA) of the crude and lintnerized starch was carried out by using thermogravimetric analyzer (TGA4000 PerkinElmer). The Thermal stabilities of the starches were evaluated according to the method employed by Zhang et al., (2015) and Zhang and Tsao, (2016). Starch samples (6 mg) each were dissolved in 18mg of distilled water, placed in alumina pans in a nitrogen atmosphere (gas flow rate 50 ml/min). and left to stand for 70 minutes. The pans were heated from 30°C to 600°C at the rate of 20°C/min. The total weight loss was recorded with the increase in temperature. And the thermographs obtained.

### Physicochemical Properties of Crude and Lintnerized Starches

The physicochemical properties of the starch samples were tested for water holding capacity, swelling power, solubility and paste clarity.

### Determination of Water holding Capacity (WHC)

The water holding capacity of the starch was determined using the method of Yadav et al. (2018). The starch suspension was prepared by dissolving 1 g of starch in 15 ml

distilled water, agitated for 2 mins and centrifuged at 1500 rpm for 10 mins. The supernatant was decanted and wet starch was weighed. The amount of water (g/g) absorbed by the sample was reported as water holding capacity as shown in equation 3.1. The analysis was carried out in triplicate, mean and standard deviation of the result was determined using IBM statistical package for social sciences (SPSS) version 23. 
$$\text{WHC (g/g)} = \frac{\text{weight of wet starch} - \text{weight of dry starch sample (g)}}{\text{weight of dry starch sample (g)}} \quad \text{Eqn. 2}$$

### Determination of Solubility

Solubility properties of the starch samples were determined according to the method described by Nuwamanya et al., (2019). Starch suspension (0.5 g) was dispersed in distilled water (15 ml) and boiled in a water bath at temperatures of 90°C (Immediately after the gelatinization temperature), with continuous stirring for 30 min. Subsequently, the sample was cooled to room temperature. After cooling, it was centrifuged at 1500 rpm for 15 mins, the supernatant was decanted and dried in an oven at 105°C for 2 hrs, and the weight of the sediment was also noted. The total amount of soluble starch (carbohydrates) was calculated using equation 3.2 (Nuwamanya et al., (2019)). The analysis was carried out in triplicate, mean and standard deviation of the result was determined using IBM statistical package for social sciences (SPSS) version 23.

$$\text{Solubility (\%)} = \frac{\text{weight of soluble starch}}{\text{weight of dry sample}} \times 100 \quad \text{Eqn. 3}$$

### Determination of Swelling Power

The swelling power of the starch samples was determined following the Shrestha et al., (2018) method as described by Raungrusmee, and Anal., (2019). The starch sample (0.5 g) was dispersed in distilled water (15 ml) in a centrifuge tube and heated in a water bath at 90°C (Immediately after the gelatinization temperature), for 30 min with continuous stirring. After cooling to room temperature, it was centrifuged at 1500 rpm for 15 mins, the supernatant was decanted and the weight of the residue was determined. The swelling power was calculated using the following equations (Raungrusmee, and Anal., (2019)). The analysis was carried out in triplicate, mean and standard deviation of the result was determined using IBM statistical package for social sciences (SPSS) version 23.

$$\text{Swelling power (g/g)} = \frac{\text{weight of swollen starch(g)}}{\text{weight of dry sample (g)}} \quad \text{Eqn. 4}$$

### Paste clarity

Paste clarity of both crude and lintnerized starches were determined according to Kalu et al., (2022) and Nuwamanya, et al., (2011). A 1% aqueous solution of starch was boiled at 93°C with repeated shaking for 30 min. The solution was transferred into a cuvette after cooling and absorbance was measured at 650 nm using a UV spectrophotometer.

The paste clarity is calculated using equation 3.4

$$\% \text{ PC} = 10^{(-A)} \times 100 \quad \text{Eqn. 5}$$

## RESULTS AND DISCUSSION

### FTIR Spectroscopy

The FTIR spectra of the crude and lintnerized starches is presented in table 1. From the FTIR spectra, it can be observed that all the crude starches have four specific absorption peaks; A broad peaks at 3291, 3302 3298 and 3300 $\text{cm}^{-1}$  for Unsoaked crude starch (U-Cs), 24 hrs soaked crude starch (S24-Cs) and 48 hrs soaked crude starch (S48 -Cs) respectively (Fig. 4). These bands are associated with the presence of a hydroxyl group in starch. (Na et al., 2021). while the aliphatic C-H stretch is observed as a sharp peak at 2929, 2827, 2933 and 2927,  $\text{cm}^{-1}$  (Dastidar and Netravali, 2012). The peak at 1636  $\text{cm}^{-1}$  is assigned to the water adsorbed by starch molecules. and the peaks at 997 and 995  $\text{cm}^{-1}$  are attributed to the glucopyranose ring C-O stretching vibrations. (Figure 4.10). Lintnerized starches (Fig. 5.6 and 7) have a disappearing band C-OH stretching absorption peaks at 3302 $\text{cm}^{-1}$ , and an additional band at 1723 $\text{cm}^{-1}$  which is attributed to the C=O stretch of conjugated ester, another peak at 1220 $\text{cm}^{-1}$  attributed to C-O-C stretch of ester (Dastidar and

Netravali, 2012). The broad peak at 2117, 2089  $\text{cm}^{-1}$  is attributed to the stretching of hydrogen bonded hydroxyl groups. Hydroxyl, O-H, deformations and C-O stretching modes show peaks at 1420, 1388  $\text{cm}^{-1}$  and 1340, 1302  $\text{cm}^{-1}$ , respectively.

On comparing the spectra of crude and lintnerized starches, shown in figure 4.10-4.13, the presence of ester bond can be confirmed by the presence of carbonyl (C=O) peak in all the lintnerized starches that is observed at 1725, 1727  $\text{cm}^{-1}$ . It was further noticed that the carbonyl stretching peak for lintnerized starches appeared at 1664  $\text{cm}^{-1}$ . These results are similar to those observed by Sauperl and Stana-Kleinschek (2010) in which the carbonyl peak for unreacted 1,2,3,4-butanetetracarboxylic acid appeared at 1701  $\text{cm}^{-1}$  and shifted to 1725  $\text{cm}^{-1}$  after ester formation (Sauperl & Stana-Kleinschek, 2010; Yang, Lu, & Lickfield, 2002). Mathew and Abraham (2007) in Dastidar and Netravali, (2012) reported the esterification of crude starch with citric acid which showed the presence of carbonyl peak in the FTIR spectrum at around 1727  $\text{cm}^{-1}$  which was absent in the FTIR spectra of the crude starches.

**Table 1: FTIR Result for Crude and Lintnerized Starches**

| Sample | Observed Peak | Interpretation                         |
|--------|---------------|--|
| Crude  | 3285          | O-H stretch of alcohols (broad band)   |
|        | 2929          | C-H stretch of aliphatic ring          |
|        | 1638          | H-OH stretch of surface absorbed water |
|        | 997           | C-O stretch of polysaccharide ring     |
| 1M     | 3302          | O-H stretch of alcohols (narrow)       |
|        | 2827          | C-H stretch of aliphatic ring          |
|        | 1723          | C=O stretch of conjugated ester        |
|        | 1220          | C-O-C stretch of ester                 |
|        | 1101, 1075    | C-O stretch of polysaccharides/starch  |
|        | 997           | C-O stretch of polysaccharide ring     |
| 3 M    | 3298          | O-H stretch of alcohols                |
|        | 2933,2827     | C-H stretch of aliphatic ring          |
|        | 1727, 1723    | C=O stretch of conjugated ester        |
|        | 997           | C-O stretch                            |
| 5 M    | 3300,         | O-H stretch of alcohols                |
|        | 2924          | C-H stretch of aliphatic ring          |
|        | 1727          | C=O stretch of conjugated ester        |
|        | 1220          | C-O-C stretch of ester                 |
|        | 995           | C-O stretch                            |

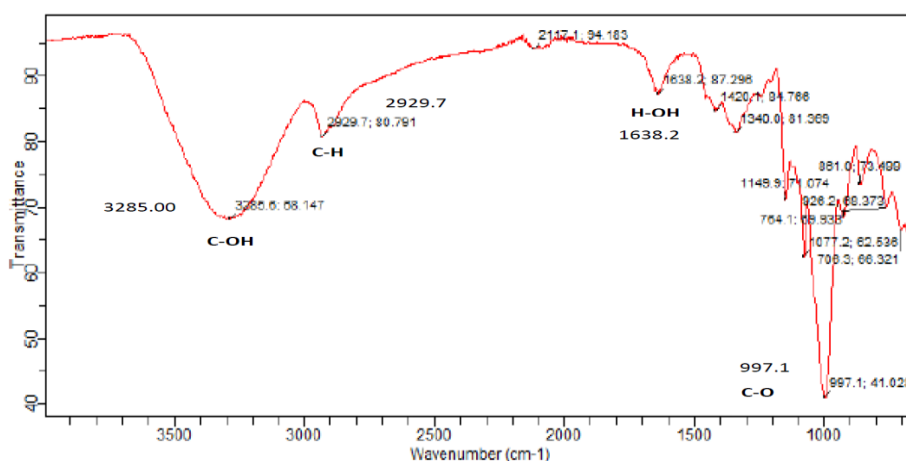


Figure 4. FTIR Spectra of Crude Starch

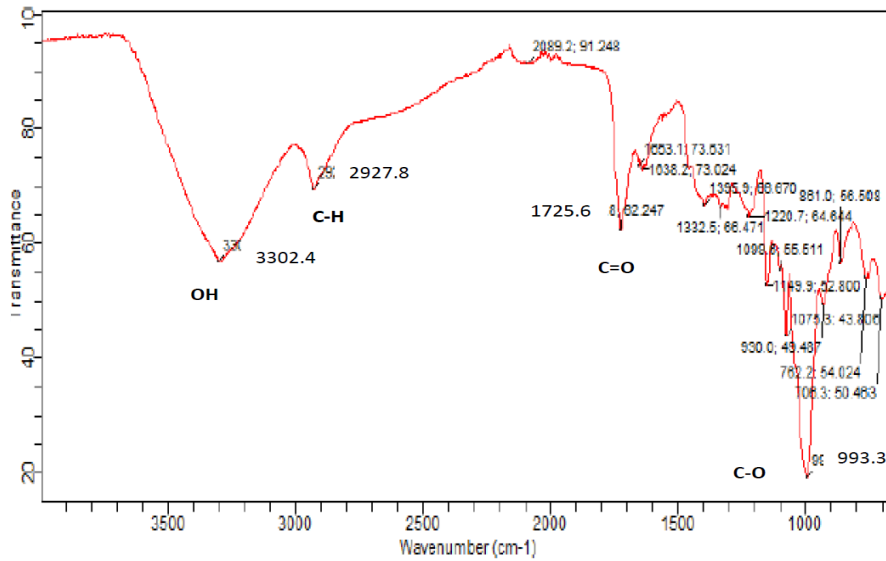


Figure 5. FTIR Spectra of 1 M Lintnerized Starch

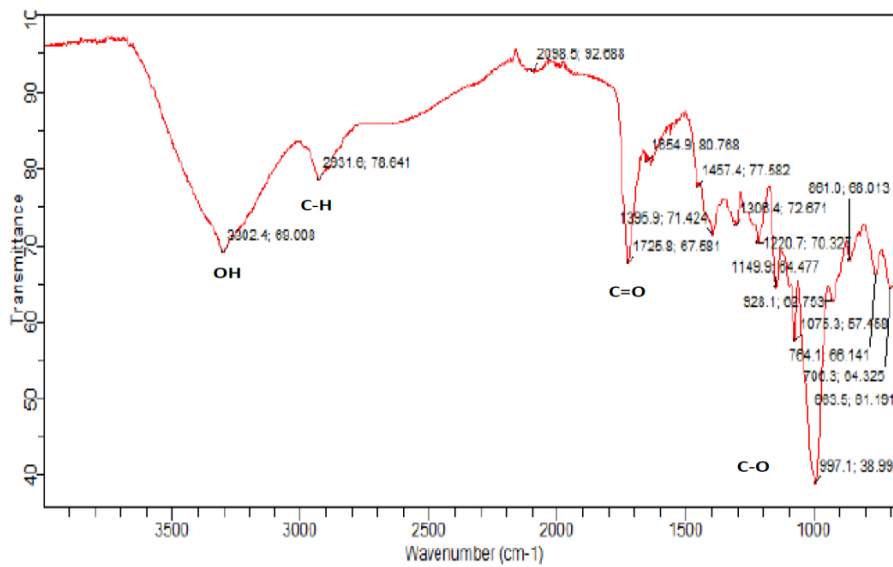


Figure 6. FTIR Spectra of 3 M Lintnerized Starch

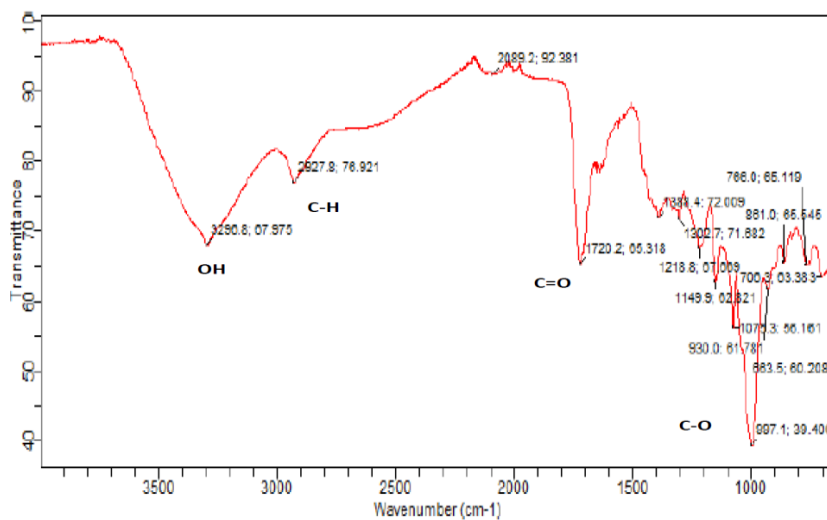


Figure 7. FTIR Spectra of 5 M Lintnerized Starch

### Degree of Substitution

The degree of substitution (DS) of the lintnerized starch was presented in Figure 13. The values for the degree of substitution for the lintnerized starches range between 0.5427, 0.7047 and 0.7817 for the 1M, 3M, and 5M respectively. From the results obtained, it was discovered that the degree of substitution increases with increase in citric acid concentration. This is similar to the result presented by Kalu *et al.*, (2022). The DS of a starch is defined as the number of hydroxyl groups substituted per d-glucopyranosyl ring (Dastidar and Netraevali 2012)). Since each ring possesses

three hydroxyl groups, the maximum DS possible is 3. However, the primary hydroxyl group (C-6) is much more reactive than the two secondary hydroxyl groups (C-2 and C-3) due to steric hindrance (Mathew & Abraham, 2007). The DS is affected by various factors like source of starch, amylose and amylopectin content, reactant concentration, reaction time and temperature (Mathew & Abraham, 2007; Xu, Miladinov, & Hanna, 2004; Zhu, Zhang, & Lai, 2007). Also, the DS is dependent on the extent of modification of starch which is dependent on the end-user's applications.

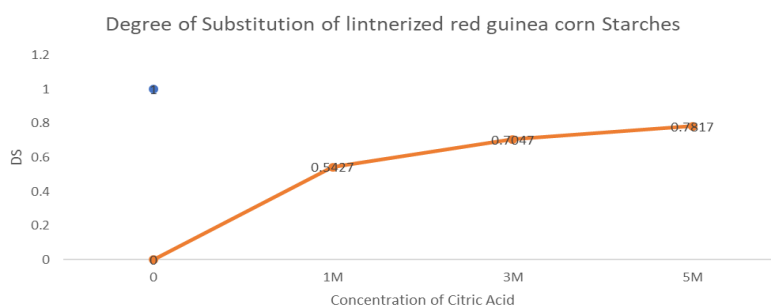


Figure 8. Degree of Substitution of Lintnerized Red Guinea Corn Starch

### Thermogravimetric Analysis

The result of the TGA analysis (Tables 2) shows that crude starches manifest excellent thermal stability, with minimal weight loss. While the lintnerized starches show higher total weight loss of 5% and lower final residue of 10% in U-M1-RGCS and 13% in U-M5-RGCS. This can be attributed to the fact that citric acid treatment purifies the starch by removing non-starch components and breakdown less crystalline regions, making the final cleaner (less inorganic ash) and making the degradation step more complete. The initial weight loss falls between 2.0% (U-Cs-RGCS) to 5.0% (U-M1-RGCS and U-M5-RGCS), a rapid second weight loss was observed from 200 to 350 °C (as shown in fig. 9), which

is associated with starch pyrolysis. Weight loss above 350 °C is associated with the decomposition of lignin and cellulose derivatives present in starch (Khawas and Deka (2017)). At 450 °C, weight loss of lintnerized starch is higher than the crude starches. The thermal decomposition of starch is mainly influenced by amylose and amylopectin contents. An increase in the amylose to amylopectin ratio of the starch can decrease the thermal decomposition temperature as amylopectin chains require more energy to breakdown due to high molecular weight. The starch with more amylose content was reported to have low thermal stability (Altayan, et al., 2021 in Ali et al., 2023).

Table 2: Thermal Decomposition of Crude and Lintnerized Starches

| Sample    | Initial Wt loss (%) Moisture | Stage II loss (%) Pyrolysis | Stage III loss (%) Carbonization | Total Weight loss (%) | Residual weight (%) |
|-----------|------------------------------|-----------------------------|----------------------------------|-----------------------|---------------------|
| U-Cs-RGCS | 2.0                          | 78.0                        | 9.0                              | 89.0                  | 11.0                |
| U-M1-RGCS | 5.0                          | 74.0                        | 11.0                             | 90.0                  | 10.0                |
| U-M5-RGCS | 5.0                          | 73.0                        | 9.0                              | 87.0                  | 13.0                |

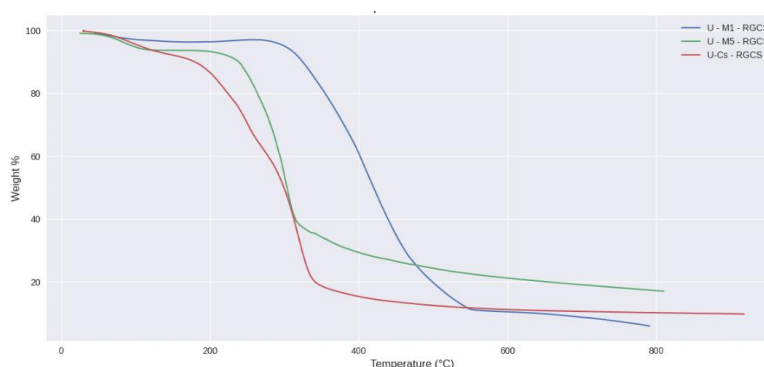


Figure 9: Thermogram of Crude and Lintnerized Starches

### Paste Clarity

The result of paste clarity (PC) is presented in Figure 10. From the result, crude red guinea corn starch has % PC of 23%, 28% and 41% for unsoaked (U-Cs), 24 hrs soaking (S24-Cs) and 48 hrs soaking (S48-Cs) respectively. 1 M lintnerized

starch have values of 31%, 29% and 32% for U-M3, S24-M3 and S48-M3 respectively. While 3 M lintnerized starch have values of 50%, 48% and 43% for U-M1, S24-M1 and S48-M1 respectively. And 5 M lintnerized starch have values of 33%, 34% and 59% for U-M5, S24-M5 and S48-M5 respectively.

Clarity of starch has a direct relationship with the concentration of citric acid and soaking pretreatment. In this study, paste clarity rose from 23% (U-CS) to 59% (S<sub>48</sub>-M<sub>5</sub>). From the result obtained in this research, % paste clarity increases with the concentration of citric acid modification. This was also reported by KarmvIr *et al.*, (2018). In his findings; chemical modification improves paste clarity of starch. Clarity characteristics of starches are dependent on the botanical source of starch and the overall purity of the starch in case of cereal starches. However, paste clarity of starch is also influenced by the amylose and amylopectin content

especially in cereal starches (Ogunmolayusi *et al.*, 2016). Increased clarity has also been reported in cassava starch films crosslinked with citric acid, attributed to reduced retrogradation and improved transparency (Reddy & Yang, 2010). To improve paste clarity of starch, chemical modification is usually a solution according to Zhang *et al.*, (2005). In this present study, starch clarity was achieved with citric acid modification and there was corresponding rise in clarity as the concentration of citric acid increases. This property is particularly valuable in transparent gels, beverages, and edible films (Adebowale *et al.*, 2005).

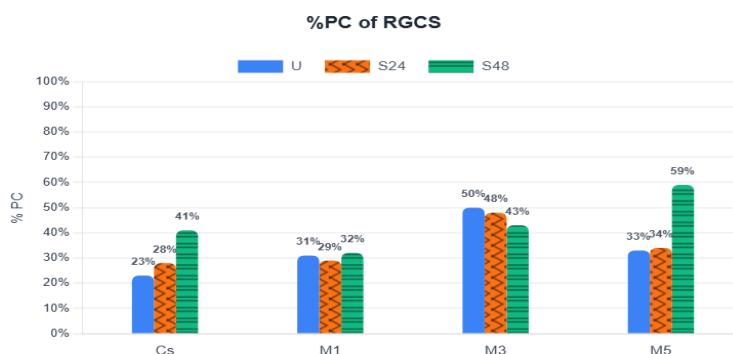


Figure 10: %PC of Red Guinea Corn Starch

### Solubility (%)

The result for the % solubility of crude and lintnerized red guinea corn starches is presented in (Figure 11) The crude starches have the solubility values of 8.00 %  $\pm$  0.00, 7.00 %  $\pm$  1.4, 10.00 %  $\pm$  0.00 for the unsoaked crude starch, (U-CS-RGCS), 24 hrs soaking crude starch (S<sub>24</sub>-Cs- RGCS) and 48 hrs soaking crude starch (S<sub>48</sub>-Cs- RGCS) respectively. While the values for 1 M citric acid lintnerization are; 20.00 %  $\pm$  4.0, 18.00 %  $\pm$  3.5, 31.33 %  $\pm$  10.1 for Unsoaked 1M modified starch (U-M1- RGCS), 24 hrs soaking-1 M modified starch (S<sub>24</sub>-M1- RGCS) and 48 hrs -1 M lintnerized starches (S<sub>48</sub>-M1- RGCS) respectively. The solubility values for 3 M citric acid lintnerization are 10.00 %  $\pm$  0.00, 20.00 %  $\pm$  4.0, 20.00 %

$\pm$  2.0 for Unsoaked 3M lintnerized starch (U-M<sub>3</sub>- RGCS), 24 hrs soaking-3 M lintnerized starch (S<sub>24</sub>-M<sub>3</sub>- RGCS) and 48 hrs -3 M lintnerized starches (S<sub>48</sub>-M<sub>3</sub>- RGCS) respectively. And the values for 5 M citric acid lintnerization are; 26.67 %  $\pm$  9.0, 30.00 %  $\pm$  3.5, 32.00 %  $\pm$  11.1 for Unsoaked 5M lintnerized starch (U-M<sub>5</sub>- RGCS), 24 hrs soaking-5 M lintnerized starch (S<sub>24</sub>-M<sub>5</sub>- RGCS) and 48 hrs -5 M modified starches (S<sub>48</sub>-M<sub>5</sub>- RGCS) respectively. From the result obtained it was discovered that crude starches, (Unsoaked, 24 hrs, and 48 hrs soaking) all have lower solubility than their lintnerized counterparts, indicating that crude starch granules are relatively intact and have resistance to water dispersion.

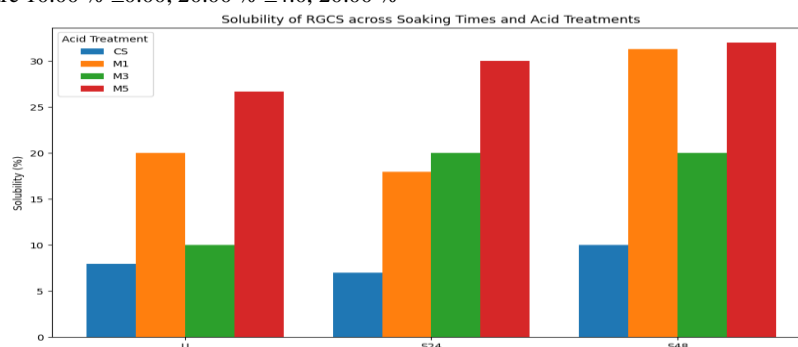


Figure 11: % Solubilities of Crude and Lintnerized RGCS Starches

### Swelling Power

The result for the swelling power of crude and lintnerized red guinea corn starches is presented in (Figure 12). Crude starches have the SP values of 10.89  $\pm$  1.13 g/g, 10.63  $\pm$  1.69 g/g, 7.80  $\pm$  0.39 g/g for the unsoaked, (U-CS-RGCS), 24 hrs soaking (S<sub>24</sub>-Cs- RGCS) and 48 hrs soaking (S<sub>48</sub>-Cs- RGCS) respectively. While the values for 1 M citric acid lintnerization are; 8.94  $\pm$  0.23 g/g, 9.23  $\pm$  0.59 g/g, 8.48  $\pm$  0.49 g/g for Unsoaked 1M lintnerized starch (U-M<sub>1</sub>- RGCS), 24 hrs soaking-1 M lintnerized starch (S<sub>24</sub>-M<sub>1</sub>- RGCS) and 48 hrs 1 M lintnerized starches (S<sub>48</sub>-M<sub>1</sub>- RGCS) respectively. The SP values for 3 M citric acid lintnerization are 8.77  $\pm$  0.28

g/g, 8.49  $\pm$  0.08 g/g and 7.98  $\pm$  0.56 g/g for Unsoaked 3M lintnerized starch (U-M<sub>3</sub>- RGCS), 24 hrs soaking-3 M lintnerized starch (S<sub>24</sub>-M<sub>3</sub>- RGCS) and 48 hrs 3 M lintnerized starches (S<sub>48</sub>-M<sub>3</sub>- RGCS) respectively. And the values for 5 M citric acid lintnerization are; 8.09  $\pm$  0.69 g/g, 8.35  $\pm$  0.06 and 5.33  $\pm$  0.35 g/g for Unsoaked 5M lintnerized starch (U-M<sub>5</sub>- RGCS), 24 hrs soaking-5 M lintnerized starch (S<sub>24</sub>-M<sub>5</sub>- RGCS) and 48 hrs 5 M lintnerized starches (S<sub>48</sub>-M<sub>5</sub>- RGCS) respectively. From the result obtained it was discovered that SP value decreases with increase in concentration of citric acid lintnerization.

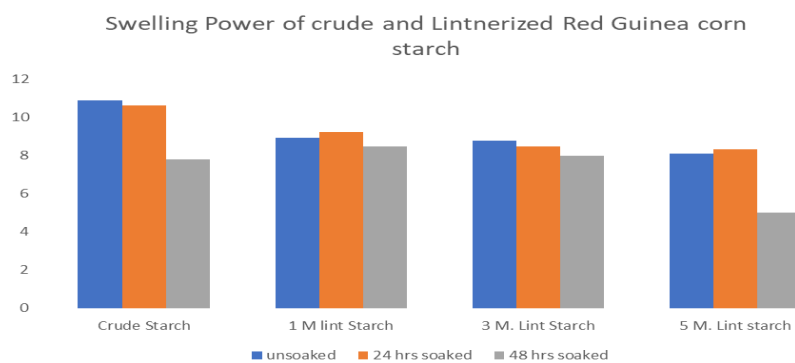


Figure 12: Swelling Power of Crude and Lintnerized Red Guinea Corn Starch

### Water Holding Capacity

The result for the water Holding Capacity (WHC) of crude and lintnerized red guinea corn starches is presented in (Figure 13). Crude starches have the WHC values of  $1.41 \pm 0.03$  g/g,  $1.09 \pm 0.02$  g/g,  $1.04 \pm 0.06$  g/g for the unsoaked starch, (U-Cs-RGCS), 24 hrs soaking (S<sub>24</sub>-Cs- RGCS) and 48 hrs soaking (S<sub>48</sub>-Cs- RGCS) respectively. While the values for 1 M citric acid lintnerization are;  $1.19 \pm 0.19$  g/g,  $1.09 \pm 0.05$  g/g,  $0.81 \pm 0.04$  g/g for Unsoaked 1M lintnerized starch (U-M<sub>1</sub>- RGCS), 24 hrs soaking-1 M lintnerized starch (S<sub>24</sub>-M<sub>1</sub>-RGCS) and 48 hrs -1 M lintnerized starches (S<sub>48</sub>-M<sub>1</sub>- RGCS) respectively. The WHC values for 3 M citric acid lintnerization are  $0.88 \pm 0.06$  g/g,  $0.79 \pm 0.13$  g/g and  $0.98 \pm 0.07$

g/g for Unsoaked 3M modified starch (U-M<sub>3</sub>- RGCS), 24 hrs soaking-3 M lintnerized starch (S<sub>24</sub>-M<sub>3</sub>- RGCS) and 48 hrs- 3 M lintnerized starches (S<sub>48</sub>-M<sub>3</sub>- RGCS) respectively. And the values for 5 M citric acid lintnerization are;  $0.73 \pm 0.04$  g/g,  $0.82 \pm 0.03$  and  $0.70 \pm 0.03$  g/g for Unsoaked 5M lintnerized starch (U-M<sub>5</sub>- RGCS), 24 hrs soaking-5 M lintnerized starch (S<sub>24</sub>-M<sub>5</sub>- RGCS) and 48 hrs -5 M lintnerized starches (S<sub>48</sub>-M<sub>5</sub>-RGCS) respectively. From the result obtained it was discovered that WHC value decreases with increase in concentration of citric acid modification. Table 3. Showed the physicochemical parameters for for the crude and lintnerized red guinea corn starches.

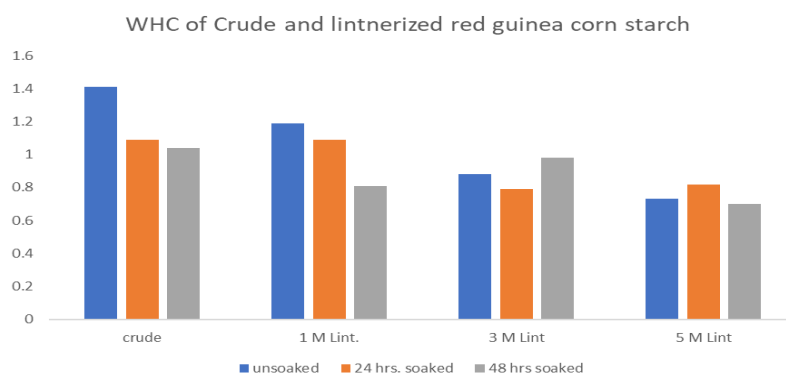


Figure 13: Water Holding Capacity of Crude and lintnerized Red Guinea Corn Starch

Table 3: Physicochemical Parameters of Red Guinea Corn Starch

| TREATMENT                       | SP (g/g)    | WHC (g/g) | % S        | % PC |
|---------------------------------|-------------|-----------|------------|------|
| U-C <sub>s</sub>                | 10.89 ±1.13 | 1.41±0.03 | 8.00±0.0   | 23   |
| S <sub>24</sub> -C <sub>s</sub> | 10.63 ±1.69 | 1.09±0.02 | 7.00±1.4   | 28   |
| S <sub>48</sub> -C <sub>s</sub> | 7.80 ±0.39  | 1.04±0.06 | 10.00±0.0  | 41   |
| U-M <sub>1</sub>                | 8.94 ±0.23  | 1.19±0.19 | 20.00±4.0  | 31   |
| S <sub>24</sub> -M <sub>1</sub> | 9.23 ±0.59  | 1.09±0.05 | 18.00±3.5  | 29   |
| S <sub>48</sub> -M <sub>1</sub> | 8.48 ±0.49  | 0.81±0.04 | 31.33±10.1 | 32   |
| U-M <sub>3</sub>                | 8.77 ±0.28  | 0.88±0.06 | 10.00±0.0  | 50   |
| S <sub>24</sub> -M <sub>3</sub> | 8.49 ±0.08  | 0.79±0.13 | 20.00±4.0  | 48   |
| S <sub>48</sub> -M <sub>3</sub> | 7.98 ±0.56  | 0.98±0.07 | 20.00±2.0  | 43   |
| U-M <sub>5</sub>                | 8.09 ±0.69  | 0.73±0.04 | 26.67±9.0  | 33   |
| S <sub>24</sub> -M <sub>5</sub> | 8.35 ±0.06  | 0.82±0.03 | 30.00±3.5  | 34   |
| S <sub>48</sub> -M <sub>5</sub> | 5.33 ±0.35  | 0.70±0.03 | 32.00±11.1 | 59   |

### CONCLUSION

This study revealed significant changes in the physicochemical, functional properties of red guinea corn lintnerized starches. The effect of pretreatment on the citric acid lintnerization was observed on the physicochemical properties. FTIR spectroscopy analysis also showed that there

are substantial structural differences between the crude and lintnerized starches confirming esterification through the appearance of carbonyl and ester peaks. Thermogravimetry analysis (TGA) indicated reduced thermal stability in the lintnerized starches. Lintnerization of pretreated red guinea corn starches with citric acid have significant effect on

functional and structural properties like water holding capacity, swelling power and Paste clarity. This dual-stage modification strategy provides a pathway for tailoring starch properties to meet industrial demands for improved solubility and paste clarity while reducing swelling and water retention, positioning it as a viable gluten-free alternative to cereal starches, indicating its potential applications in food formulations requiring controlled viscosity, gel stability, and improved thermal resistance. Therefore, lintnerized red guinea corn starch could be a healthy and good source of starch for industrial application such as in baking, the production of children foods, and cookies.

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