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### Synthesis, Characterization and Water Swelling Properties of *Borassus Aethiopum* Starch-G-Polyacrylamide Hydrogels

Abdulkadir Abubakar<sup>\*1,2</sup>, Boryo Doris Ezekiel Amin<sup>1</sup>, Auwal Adamu Mahmoud<sup>1</sup>, Abubakar Samaila<sup>2</sup>

<sup>1</sup>Department of Chemistry, Faculty of science, Abubakar Tafawa Balewa University, PMB 0248, Bauchi, Nigeria

<sup>2</sup>Department of Chemistry, Faculty of science, Gombe State University, PMB 127, Gombe, Nigeria

#### ABSTRACT

Starch was extracted from *Borassus aethiopum* tuber using blending, filtration decantation methods. The starch-g-polyacrylamide was prepared by graft copolymerization between the *Borassus aethiopum* starch and acrylamide via solution polymerization using potassium per sulphate (KPS), as initiator and  $N^1$ ,  $N^1$ -Methylenebisacrylamide (crosslinker) respectively. The maximum water swelling percentage of the starch-g-polyacrylamide obtained at 0.2 g of initiator content was found to be 470 % with starch to the monomer ratio of 1:1. The functional group of the *Borassus aethiopum* starch, and the synthesized hydrogel were characterized by fourier transform infrared (FTIR) spectroscopy (model Cary 630 MY13270004 G8043-64002). The IR spectra of *Borassus aethiopum* starch obtained depict the presence of O-H stretching absorption band in the region of  $3286\text{ cm}^{-1}$  (broad). The C-H bond stretching at  $2933\text{ cm}^{-1}$  (medium) and absorption bands of  $1153$ ,  $1082$  and  $996\text{ cm}^{-1}$  (s) for the C-O-C bond stretching. While spectra of starch-g-polyacrylamide display N-H stretching in the region  $3500\text{--}3200\text{ cm}^{-1}$ ,  $1655$ , and  $1607\text{ cm}^{-1}$  indicated the presence of C=O stretching and N-H bending of the amide bands, respectively. This revealed the presence of  $\text{-CONH}_2$  functional group of the acrylamide before and after purification which shows all the absorption peaks of *Borassus aethiopum* starch. The raw starch and synthesized hydrogels were characterized using SEM and DSC. The SEM micrograph of the starch sample showed irregular shapes and varied particle sizes with white smooth surfaces. The SEM micrograph of the starch-g-polyacrylamide shows a very smooth surface with three dimensional networks on the SEM micrograph. The thermal stability of the *Borassus aethiopum* starch and starch-g-polyacrylamide hydrogels was also determined and found to be  $68^\circ\text{C}$  and  $75^\circ\text{C}$  respectively. This shows improvement of the thermal stability of the synthesized hydrogel. Therefore, purified *Borassus aethiopum* starch could be used in the synthesis of hydrogel.

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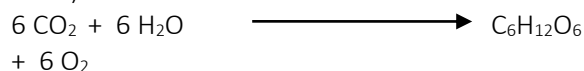
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#### KEYWORDS

Starch,  
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## 1. Introduction

Starch occurs in plants and utilized by animals. Starch is synthesized by green plants during photosynthesis a complex process in which sunlight provides the energy to convert CO<sub>2</sub> and H<sub>2</sub>O into glucose plus oxygen. Many molecules of glucose are then chemically linked through glycoside (acetal linkage) bond to form either cellulose or starch which is stored in plants (John, 2007).



Starch is the second most abundant organic compound found on the earth. Starch is a polymer of glucose in which the monosaccharide units are linked by 1 to 4-  $\alpha$ - glycoside bonds like those in maltose. Starch can be separated into amylose and amylopectin (Muazu et al., 2014). Starch is used in variety of industries which include food, textile, cosmetics, plastics, adhesives, paper and pharmaceuticals (Manek et al., 2012). Starch is also used as fillers, binders (Okunlola and Odeku, 2011) and lubricant (Muazu et al., 2014).

*Borassus aethiopum* has been described as a palm tree with huge fan shaped leaves. The various ethnic group in Nigeria like the Hausa calls it Ginginya, the Yoruba Agbon Olodu, the Ibos Ubiri and the Kanuri Kemeletu (Aguzue et al., 2013). Tangale calls Kanje (Boryo et al., 2010). Also, Arabic deleib-delieb, English Borassus palm, African fan palm, Fan palm, Palmyra palm and French Palmier ronier-rônier (Boryo et al., 2010). *Borassus* palm tree has been used in the construction of roofing, mat and rope (Ali et al., 2010). The cellulosic fibre properties are used as an alternative raw material for the manufacture of paper. The plant part is also important in the treatment of gonorrhea, dysentery and respiratory infections. The fruit (pulp), an orange to yellow fibrous tissue is rich in vitamin A, B and C (Adzinyo et al., 2015).

The uses of the root (tuber), shoot and fruits of the plant in traditional medicine for the treatment of various ailments have been reported (Sarma and Mahanta, 2000). The profitability of the plant has also been studied

(Jatau, 2008). The young germinating shoot or the hypocotyls known as Muruchi (in hausa) which appears about two months after the seeds are grown, is harvested, being very starchy, and considered of great value in terms of famine (Akiniyi et al., 2011). *Borassus aethiopum* tuber is an important source of food which is rich in carbohydrate and protein for the rural of Northern Nigeria (Boryo et al., 2010). Both young and old people consume Muruchi either raw or boiled as food and it is claimed that it enhances libido in women and has aphrodisiac properties in men (Akinniyi et al., 2010). Starch is the only qualitatively important digestible polysaccharide and has been regarded as nutritionally superior to low molecular weight carbohydrate or sugar (Aguzue et al., 2013). *Borassus aethiopum* starch was among the underutilized starch in West Africa because the full potential of the starch for synthesis of hydrogels is yet to be exploited.

Hydrogels are three-dimensional, hydrophilic, polymeric networks capable of absorbing large amounts of water or biological fluids (Shivani et al., 2015). Due to their high-water content, porosity and soft consistency, hydrogels closely simulate natural living tissue, more than any other class of synthetic biomaterial (Shivani et al., 2015). When swelled, it is soft, rubbery and resembles the living tissue (Devi et al., 2014). Hydrogels may be chemically stable or they may degrade and eventually disintegrate and dissolve at high temperature (Caló and Khutoryanskiy, 2015). Hydrogels are prepared from materials such as gelatin, polysaccharides, cross-linked polyacrylamide polymers, polyelectrolyte complexes, and polymers or copolymers derived from methacrylate esters (Muhammad et al., 2015). Hydrogels are insoluble in water and are available in dry or hydrated sheets or as a hydrated gel in drug delivery systems designed for single use (Lee et al., 2013, Buwalda et al., 2014 and Shivani et al., 2015).

In general, hydrogels can be prepared from either synthetic polymers or natural polymers (Jinlei et al., 2010). The synthetic polymers are hydrophobic in nature and chemically stronger compared to natural polymers (Ahmed, 2015).

Their mechanical strength results in slow degradation rate, but on the other hand, mechanical strength provides the durability as well (Yom *et al.*, 2014). These two opposite properties should be balanced through optimal design. Water-soluble polymers of both natural and synthetic origin are cross-linked to form hydrogels in a number of ways (Das, 2013). Linking polymer chains via chemical reaction, using ionizing radiation and physical interactions such as entanglements, electrostatics, and crystallite formation are importance (Ahmed, 2015).

Furthermore, hydrogels can be formulated in a variety of physical forms, including slabs, microparticles, nanoparticles, coatings, and films (Lee and Kim, 2011). As a result, hydrogels are commonly used in clinical practice and medicine with a wide range of applications (Slaughter *et al.*, 2009), including tissue engineering and regenerative medicine, diagnostics, cellular immobilization, separation of biomolecules or cells, and barrier materials to regulate biological adhesions (Hoare and Kohane, 2008).

There are three integral parts of the hydrogels preparation these are monomer, initiator, and cross-linker (Muhammad *et al.*, 2015). To control the heat of polymerization and the final hydrogels properties, diluents can be used, such as water or other aqueous solutions (Billmeyer, 2007). Hydrogels are usually prepared from polar monomers (Campoccia *et al.*, 1998). According to their starting materials, they can be divided into natural polymer and synthetic polymer (Chirani *et al.*, 2015).

Pourjavadi *et al.* (2004) synthesized a novel polysaccharide based superabsorbent hydrogel via graft copolymerization of acrylic acid onto Kappa carrageenan in air, through chemically crosslinking graft copolymerization of acrylic acid onto Kappa-carrageenan (KC) in the presence of crosslinker agents and a free radical initiator. Maximum water absorbency of the optimized final product was found to be 789 g/g the swelling capacity of the synthesized hydrogel was also measured in various salt solutions.

Biodegradable superabsorbent polymers were synthesized by graft copolymerization of acrylamide (AM)/itaconic acid (AI) onto cassava starch via a redox initiator system of ammonium per sulphate (APS) and N,N,N',N'-tetramethylethylenediamine (TEMED), methylenebisacrylamide as crosslinking agent, sodium bicarbonate foaming agent, a triblock copolymer of polyoxyethylene/polyoxypropylene/polyoxyethylene as a foam stabilizer. The acrylamide-to-itaconic acid ratio, the starch to monomer ratio, and concentrations of the crosslinking agent and initiator, on the water absorption of the superabsorbent polymers were investigated (Lanthong *et al.*, 2006). The swelling of starch-g-Polyacrylamide was 39 g/g while the starch-g-Polyacrylamide-co-itaconic acid with the itaconic acid content of 0.02 - 0.15 % mole gave the water swelling value in the range of 70-390 g/g (Lanthong *et al.*, 2006).

Yahaya *et al.* (2021) investigated the swelling behaviour of starch-g-Acrylic acid hydrogel and its potential application in removal of Rhodamine B and Alkali blue dyes. Acrylic acid monomer was successfully grafted on starch through free radical polymerization and potassium persulphate as an initiator, methylenebisacrylamide (MBA) as a crosslinker. The optimum swelling condition of the hydrogel was observed at pH 7.

This study tends to extract starch from *Borassus aethiopum* tuber and use in the synthesis of hydrogel for domestic and industrial uses. It also aimed at characterizing the starch and hydrogel using *fourier transform infrared (FTIR)*, SEM and DSC.

## 2. Materials and Methods

### 2.1. Materials

Material used include: magnetic stirrer and hot plate magnetic stirrer, set up nitrogen gas line, masking tape, crucible, weighing machine, measuring cylinder, thermometer, syringe, three necks round bottom flask, conical flask, Erlenmeyer flask, condenser, stop watch, glass beaker, glass rod, 100-mesh sieve aluminum

screen, tube, and e.t.c were provided by Abubakar Tafawa Balewa University, Bauchi State and Bayero University Kano, Kano State. Fresh Palmyra tubers (muruchi) were purchase from Kaltungo Market Gombe State, Nigeria.

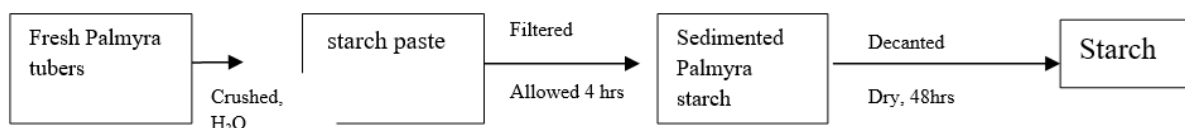
### 2.1.1 Chemicals and reagents

In this research, the *Borassus aethiopum* starch was extracted in the Laboratory of Department of Chemistry, Abubakar Tafawa Balewa University, Bauchi. Analytical grade acrylamide (Loba

Chemie Pvt Ltd, Indian), itaconic acid (Loba Chemie Pvt Ltd, Indian), nitrogen gas, potassium per sulphate, N, N<sup>1</sup>-methylenebisacrylamide (Titan Biotech Ltd, Rajasthan, and Indian), distilled water, sodium hydroxide (Titan biotech Ltd, Rajasthan, Indian), hydrochloric acid, iodine solution and ethanol were purchase from Bristol Scientific Company Limited Lagos and Ceman Scientific Ltd Kano, Nigeria. All reagents were used as received without further purification.

## 2.2. Methods

### 2.2.1. Flow chart for preparation of *Borassus aethiopum* starch.



#### *Scheme 1: Preparative flow chart for starch extraction*

### 2.2.2. Extraction of *B. aethiopum* tuber starch

*Borassus aethiopum* tuber starch was extracted by the method described by Aguzue *et al.* (2013), Muazu *et al.* (2014) and Chukwuemeka *et al.* (2018) with slight modification. Fresh Palmyra tubers was washed, peeled using stainless steel knife, crushed with blender and homogenized with distilled water. The mixture was stirred with a stirring rod and then filtered using double layered nylon cloth. The filtrate was allowed to stand for 4 hrs. It was then separated by sedimentation and decantation. The starch was dried in air on aluminum pan at room temperature for 48 hrs. Finally, a starch powder was obtained (Scheme 1).

was stirred at 250 rpm under heating within the temperature range of  $80 \pm 2$  °C for 30 min under the nitrogen atmosphere and homogeneous, soft and opaque paste gelatinized starch was formed. The gelatinized starch was allowed to cool to 45 °C. The mixture of water (50 cm<sup>3</sup>) containing acrylamide (2.0 g) and potassium per sulphate (0.1 g) was added into the reaction vessel. The reaction mixture was stirred under nitrogen atmosphere for 30 min. The reaction product was precipitated with ethanol and dried at 65 °C (Lanthong *et al.*, 2006). The final product (Hydrogel) was stored at room temperature. The procedure was repeated for different initiator (potassium per sulphate) content.

### 2.2.3. Preparation of starch-g-polyacrylamide

The method described by Lanthong *et al.* (2006), was adopted, in which the starch (2.0 g) was mixed with 25 cm<sup>3</sup> of distilled water in a 250 cm<sup>3</sup> three necked round bottom flask. The system

#### 2.2.3.1 Water Extraction (removal of free polymers)

The dried product (1.0 g in powdered form) (W<sub>1</sub>) obtained was stirred in 150 cm<sup>3</sup> of distilled water at room temperature for 24 hrs using magnetic stirrer. The grafted copolymer was separated by

filtration. It was then washed with distilled water and filtered to obtain another grafted copolymer (hydrogel). It was then dehydrated with ethanol to obtain the fine precipitate. The material was dried in an oven at 65 °C for 24 hrs and it was weighed ( $W_2$ ) using analytical balance. The final product (hydrogel) was again dried in an oven at 65 °C for 24 hrs and ground into fine powder.

## 2.3. Characterization

### 2.3.1. Determination of functional group of the hydrogels by FTIR spectroscopy analysis

The functional group of purified *Borassus aethiopum* starch, and starch-g-polyacrylamide hydrogels were examined by FTIR spectroscopy (Cary 630 MY13270004 G8043-64002). The spectra of the prepared starch and hydrogel were recorded using spectrometer. All the spectra (32 scans at 8.0 cm<sup>-1</sup>) were recorded at the range of 4000-650 cm<sup>-1</sup> at 25 °C (Lanthong et al., 2006).

### 2.3.2. Determination of water swelling percentage of the hydrogels

Water swelling percentage (absorption capacity) of dry hydrogel was determined at ambient temperature using distilled water until the hydrogel was completely swollen (Lanthong et al., 2006) the swelling measurements of the hydrogel was carried out in distilled water at room temperature. 100 ml of distilled water was added to 0.1 g of the dried grafted copolymer in 250 cm<sup>3</sup> glass beaker with a glass cover. The grafted copolymer was allowed to swell for 24 hrs. The fully swollen gel was then separated from water by filtration using a 100-mesh sieve aluminum screen for 1hr at room temperature and the swollen hydrogel was weighed. The water absorption was calculated as in the Equation (1). The water swelling percentage was determined repeatedly three times for each sample (Lanthong et al., 2006).

$$\text{Water absorbency}(Q) = \frac{W_1 - W_0}{W_0} \times 100$$

.... Equation (1)

Where  $W_0$  is the weight of the dry graft-copolymer (g),

$W_1$  is the weight of the water swollen graft-copolymer (g) Lanthong et al., 2006).

### 2.3.3. Determination of the surface morphology using SEM analysis

The method described by Hamma (2018) was adopted. The purified *Borassus aethiopum* starch and starch-g-polyacrylamide hydrogels were placed separately on double adhesive plate which was on a sample stub. It was coated with a sputter coater by quorum technologies model Q150R, with 5nm of gold. It was taken to the chamber of SEM machine where it was viewed via NaVCaM for focusing and little adjustment. It was then transferred to SEM mode. It was focused and brightness contrasting was automatically adjusted and afterward the morphology of different magnifications was stored in a USB stick.

### 2.3.4. Determination of the thermal stability using DSC analysis

The samples *Borassus aethiopum* starch and starch-hydrogel were separately and thoroughly dried in the solid state to avoid damaging the equipment. 2.0 g of each of the sample was weighed in Tzero hermetic aluminum pan. The lid was placed on the pan. The covered pan was put in the blue holder. It was then ensured that the lid was flush with the top of the holder. The handle was pulled down, which would press and sealed the pan and the lid together. The procedure was repeated with an empty pan to be used as a reference. The power was then turn on; the information was generated at TA universal analysis on the desktop of the computer. The analysis was carried out using DSC (model TA Q-20).

## 3. Results and Discussion

### 3.1. Characterization of *B. aethiopum* tuber starch and the hydrogel

The starch and hydrogel obtained was characterized using FTIR, SEM and DSC analysis.

3.1.1. Absorption bands and Functional groups for starch and starch-g-polyacrylamide.

The spectra for starch and starch-g-polyacrylamide hydrogels after purification are presented in Table 1. The IR spectra of *Borassus aethiopum* starch obtained in Table 1 depict the O-H stretching

absorption band in the region of 3288 cm<sup>-1</sup> (broad). The C-H bond stretching at 2933 cm<sup>-1</sup> (medium) and absorption bands of 1153, 1082 and 996 cm<sup>-1</sup> (s) for the C-O-C bond stretching (a triplet peak of starch). The IR spectra of starch-g-polyacrylamide presented in Table 1 display N-H stretching absorption band at 3339 cm<sup>-1</sup>.

Table 1: Absorption bands and functional groups of starch and starch-g-poly acrylamide

Standard IR absorption frequencies (cm <sup>-1</sup> ) for common functional groups		Absorption bands (cm <sup>-1</sup> ) in	
		Starch-g-polyacrylamide	Starch
O-H (Carboxylic acid) 3500-2400			
O-H (Alcohol) 3400-3200		3193	3288
N-H (Amide 1°) 3500-3200		3339	
N-H (Amide 2°) 3500-3200		3312	
C-H (sp <sup>3</sup> ) below 3000		2929	2933
C=O (Amide) 1680-1640		1655	
N-H (Bending)		1454	
N-H Out of plane bend (symmetric) 1455-1420		1421	
N-H Out of plane bend (asymmetric) 770-670		668	
-C-N (Stretching) 1450-1405		1412	
C-O-C (Ether) 1250-1150		1153	1153
C-O (Ether) 1150-1050		1112	
C-O (Alcohol) 1260-1050		1082	1082

The absorption band at 1655, and 1607 cm<sup>-1</sup> indicate the C=O stretching and N-H bending of the amide bands, respectively, which indicate the presence of —CONH<sub>2</sub> group in the acrylamide both before and after water extractions give all the absorption peaks of *Borassus aethiopum* starch.

Furthermore, the peak at 1412 and 713 cm<sup>-1</sup> is for the —C—N stretching and the weak band N—H out of plane bending respectively which is the characteristics absorption band of the amide. After the acid hydrolysis, the following absorption bands confirm the presence of



polyacrylamide as follows: The NH stretching  $O=C-NH$  (amide band), the  $C=O$  stretching, the NH bending and at  $1454\text{ cm}^{-1}$  for the  $-C-N$  stretching and the NH out of plane bending at  $668\text{ cm}^{-1}$ . There is a small characteristic peak found at  $2326\text{ cm}^{-1}$  which is the  $-C-N$  peak, an intermediate peak from the acid hydrolysis of the amide to carboxylic group. This result is at par with the work of (Sadeghi, 2012) and (Lanthong et al., 2006). Observed the peak displayed at the  $2330\text{ cm}^{-1}$  indicate the effect of acid conversion of carboxyamide to carboxylic acid through cyano-complex formation.

### 3.1.2. Effects of initiator contents on the water swelling (%) of starch-g-polyacrylamide hydrogels

The effects of initiator (Potassium per sulphate) contents on the water swelling (%) of starch-g-polyacrylamide hydrogels are shown in Figure 1.

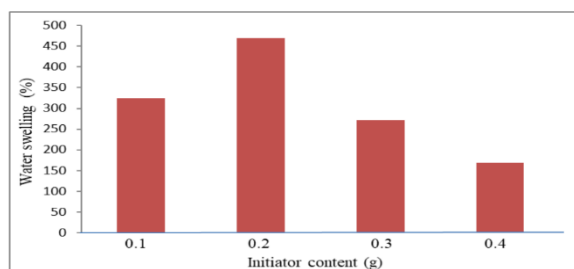


Figure 1: Effects of initiator contents on the water swelling (%) of starch-g-polyacrylamide hydrogels

The initiator (potassium per sulphate) content at 0.1 g when all parameters (starch, acrylamide and crosslinker) are kept constant, the water swelling (%) of the Starch-g-polyacrylamide hydrogel was found to be 324 (%). Increasing the initiator content from 0.1 to 0.2 g, the water swelling (%) of the synthesized hydrogel suddenly increased to 470 (%).

As the initiator content increase to 0.3 and 0.4 g the water swelling (%) of the synthesized hydrogel drastically decrease to 271 and 169 (%) respectively. Therefore, the water swelling (%) of the synthesized hydrogels increased with increasing initiator content up to 0.2 g, which gave the optimum water swelling (470 %) as shown in Figure 1. The increase of water swelling (%) observed at 0.1 and 0.2 g may be due to

increase in the active sites on the starch backbone arising from the attack of the sulfate anion radical (Potassium per sulphate). The increase in the active sites on the starch backbone may have led to increase of grafting monomers onto the starch backbone. Another possible reason for the observed increase initially may be due to the nature of the substrate which is carbohydrate in this present work. This can be confirmed from the work of Sadeghi (2012) who used carrageenan composite and observed similar increase from 160 to 240 % for the initiator concentration of 0.005 - 0.039 mol/L respectively. Sadeghi (2012) suggested that the increase in water swelling percentage may be due to imperfection of the polymer networks obtained from high-initiator polymerization systems.

The decrease of water swelling (%) with increase initiator content from 0.3 to 0.4 g as observed in Figure 1 may be due to an increase in number or degree of crosslink (intermolecular bond) within the hydrogel molecule. Another reason may be that as the initiator content increase, it led to increase in number of produced radicals favouring the terminating steps via bimolecular collision and free radical degradation of the starch substrate (Sadeghi, 2012).

In addition, the predominance of homo polymerization over grafting and molecular weight loss of the synthetic part of the polymer network, resulted in the observed trend as initiator content increase (Sadeghi, 2012).

### 3.2. Morphology of *B. aethiopum* starch

The SEM micrograph of extracted *Borassus aethiopum* starch is illustrated in Plate I

The starch granules (Plate I) show an irregular shape and different particle sizes with white smooth surface. There is porous space in between the starch granules which is responsible for the absorption of biological fluid or water. This result is in agreement with the work Lanthong et al. (2006) and Ahmad et al. (2014). Found that cassava starch has irregular shape, round and varied particle sizes with smooth surface.

The SEM micrograph of the starch sample shows the presence of starch granule from the small to large sizes. The granule surface of the starch appeared to be smooth with no sign of any fissure; this result is at par with the work of Huang (2009) and Zhu *et al.* (2011). The study revealed that guar gum and potato starches granules were found to be polygonal, round and irregular shape. Single individual granules and compound granules were also observed in the SEM micrograph. According to Newman *et al.* (2007) residual proteins might create slight gelatinization over the surface of starch granule to adhere together to form a compound granule. This might be used as fat substitute in food industry (Jane *et al.*, 1992).

### 3.2.1 Effects of initiator contents (KPS) on the SEM micrograph of starch-g-polyacrylamide hydrogels

The effects of initiator contents (potassium persulphate) on the SEM micrograph of starch-g-polyacrylamide hydrogels are displayed in Plate II. The SEM micrograph of the starch-g-polyacrylamide in Plate IIa which contains 0.1 g of  $K_2S_2O_8$  shows a very smooth surface with three dimensional networks (crosslink networks) on the SEM micrograph. This is due to grafted monomer and cross linking between the starch and acrylamide. It was confirmed by the FTIR analysis in Table 1, where the results displayed N–H stretching absorption band in the region  $3500\text{--}3200\text{ cm}^{-1}$ , C=O ( $1655\text{ cm}^{-1}$ ) stretching and N–H ( $1607\text{ cm}^{-1}$ ) bending of the amide bands, respectively which indicate the presence of —CONH<sub>2</sub> group in the acrylamide.

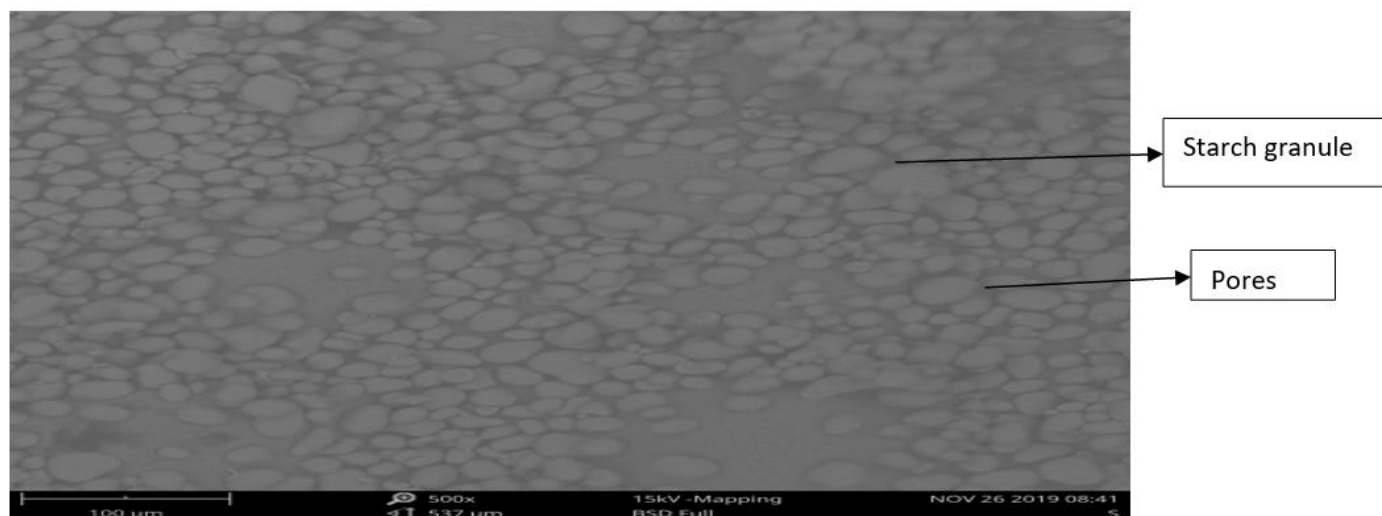


Plate I: SEM micrograph of raw starch obtained from *Borassus aethiopum*.



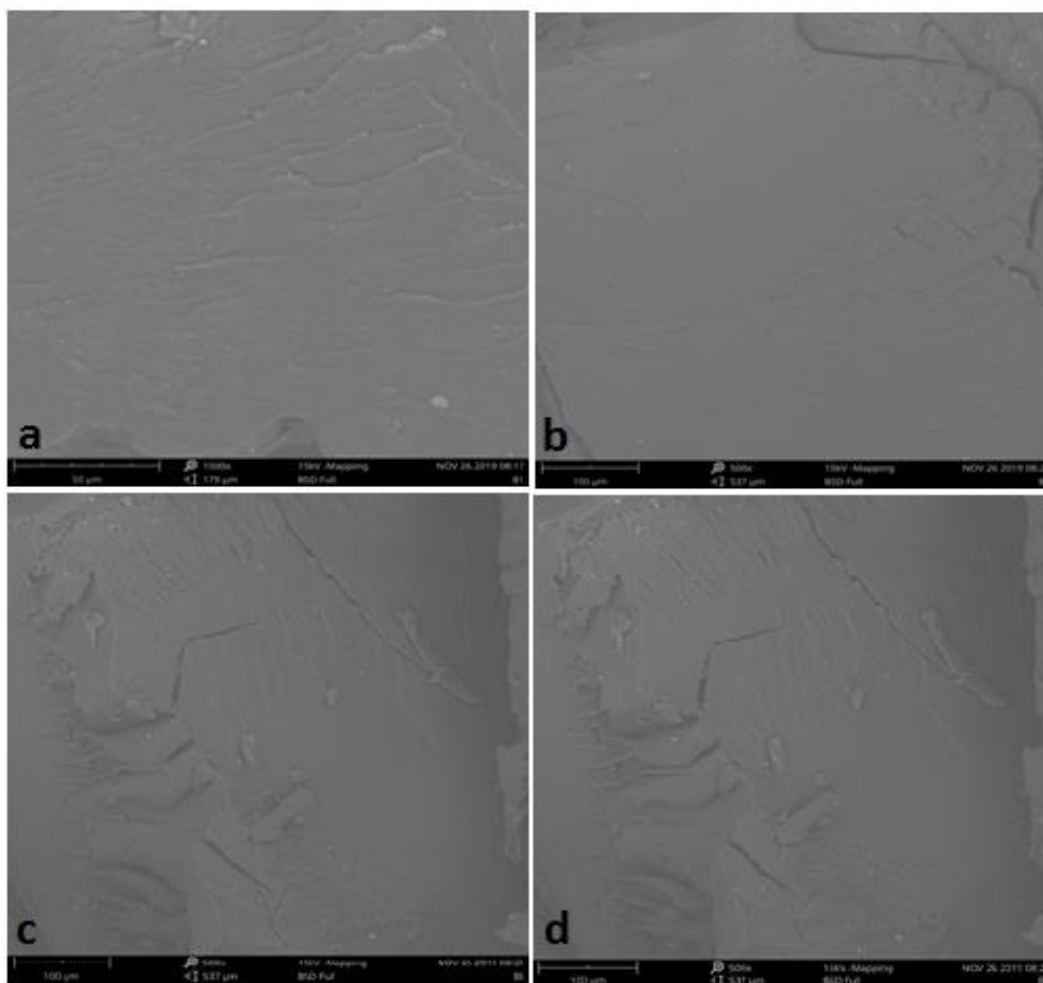


Plate II: Effects of  $K_2S_2O_8$  (initiator contents) on the morphology of starch-g-polyacrylamide hydrogels.

**Keys:** (a) 0.1 g of  $K_2S_2O_8$  (initiator contents)

(b) 0.2 g of  $K_2S_2O_8$  (initiator contents)

(c) 0.3 g of  $K_2S_2O_8$  (initiator contents)

(d) 0.4 g of  $K_2S_2O_8$  (initiator contents)

The water swelling % of starch-g-polyacrylamide hydrogel was found to be 169 % Figure 1. Therefore, the water swelling percentage of the starch-g-polyacrylamide hydrogel contains 0.1 g of  $K_2S_2O_8$  double to that of the starch-hydrogel (without acrylamide) which was found to be 95 %. The appearance of the crosslink networks on the SEM micrograph of starch-g-polyacrylamide may be due to the presence of the monomer (0.1 g of acrylamide) that has been grafted to the starch backbone. This is in agreement with the work of Lanthong *et al.* (2006) which revealed that acrylamide is water soluble and can provide

polymer chains with amide group. The side chains may increase the hydrophilic character of the graft copolymer.

Plate IIb shows the SEM micrograph of starch-g-polyacrylamide which contains 0.2 g of the initiator content. The micrograph shows relatively high crosslinking density more than that of Plate IIa (starch-g-polyacrylamide which contains 0.1 g of  $K_2S_2O_8$ ) with a very few fissured and cracks (that implies little destruction) on the surface morphology of the hydrogel. The little destruction is due to increase in initiator content from 0.1 g to 2.0 g which increase the crosslink

networks by abstraction of hydrogen on the starch backbone. This implies that the more the initiator content the higher the active chain. This result agrees with the work of Lanthong et al, (2006) and Hamma (2018). It shows that, as the initiator content increase the grafting efficiency tends to increase which leads to increase in the grafting of the monomer on the starch backbone. This also agrees with the water swelling percentage in Figure 1 of the starch-g-polyacrylamide hydrogel that contains 0.2 g of  $K_2S_2O_8$  which was greater than that of 0.1 g of  $K_2S_2O_8$  were found to be 324 % and 271 % respectively.

Plate IIc shows SEM micrograph of starch-g-polyacrylamide hydrogel which contain 0.3 g of  $K_2S_2O_8$ . It could be observed that there is evidence of increased destruction in the morphology of the hydrogel where more cracks are seen on the starch-g-polyacrylamide hydrogels that contain 0.3 g of  $K_2S_2O_8$ . This may be due to increase in the initiator content from 0.2 g to 0.3 g which led to extensive crosslink networks on the starch-g-polyacrylamide hydrogel. Extensive crosslink networks on the hydrogel results in fissuring and cracking of the hydrogel. The result agrees with Sadeghi (2012)

research work which shows that as the initiator content increase the grafting efficiency (crosslink networks) tends to increase and reached an optimum value which decrease. The water swelling capacity also decreases from 470 % to 271 % as shown in Figure 1.

Plate II d shows SEM micrograph of starch-g-polyacrylamide hydrogel that contain 0.4 g of  $K_2S_2O_8$ . This hydrogel have similar morphological character to that in Plate IIc. The only difference between them is the water swelling (%) of the hydrogels which was found to be 169 % as displayed in Figure 1. Finally, the optimum degree of crosslink network in this work was observed at 0.2 g of potassium per sulphate (KPS).

3.3 DSC Analysis

This is one of the most important properties of starch that must be considered in order to ascertain whether the starch has a well-defined heat capacity. The analysis measures the heat changes during thermal denaturing of the starch i.e. it measures the melting point ( $T_m$ ) of the starch. The thermal stability of the synthesized starch was characterized using the differential scanning Calorimetry (DSC 1 Star System-Mittler) with magnification of 350.

Table 2: Thermal stability of *Borassus aethiopum* tuber starch

Materials	Thermal stability (°C)
Raw starch	68

Table 2 shows the thermal stability of the Raw-Starch. The maximum temperature of starch was found to be 68 °C. This means that, the extracted starch has well defined heat capacity of 68 °C. Therefore, above 68 °C the starch would be thermally degraded and dissolve or melt (viscous flow).

**Effects of initiator contents on the thermal stability of starch-g-polyacrylamide hydrogel**

The effects of initiator contents on the thermal stability of starch-g-polyacrylamide hydrogel are presented in Table 3. Table 3 shows the thermal stability of the Starch-g-polyacrylamide with initiator content of 0.1 g as thermally stable at 75

°C. The thermal stability of starch-g-polyacrylamide is greater than that of raw starch and starch-hydrogel which was found to be 68 °C and 75 °C respectively. This is due to the presence of the initiator (potassium per sulphate) content in starch-g-polyacrylamide which increases the thermal stability from 68 °C to 75 °C.

**Table 3: Effects of initiator contents on the thermal stability of starch-g- polyacrylamide hydrogel**

Initiator content (g)	Thermal stability (°C)
0.00	68
0.10	75
0.20	68
0.30	68
0.40	68

It was observed that, as the initiator content increased from 0.1 g to 2.0 g, the thermal stability of the Starch-g-polyacrylamide decrease from 75 °C to 68 °C. As the initiator content increases the thermal stability become constant at a value of 68 °C. Therefore, 0.1 g of the initiator (potassium per sulphate) content was found to be the optimum thermal stability 75 °C of the synthesized Starch-g-polyacrylamide hydrogel.

**4. CONCLUSION**

The aim was to extract *B. aethiopum* starch and characterized hydrogel produce by graft copolymerization of the *B. aethiopum* starch-g-polyacrylamide hydrogels. The Starch-g-polyacrylamide was prepared; the maximum water absorption capacity of the hydrogel was determined and was found to be 470 %. The optimum condition of the initiator content that gave the highest water absorption capacity was found to be 0.2 g.

The FTIR spectra of starch-g-polyacrylamide display N–H stretching in the region 3500-3200 cm<sup>-1</sup>, 1655, and 1607 cm<sup>-1</sup> indicate the C=O stretching and N–H bending of the amide bands,

respectively, which indicate the presence of — CONH<sub>2</sub> group in the acrylamide.

The morphology of the hydrogels was observed to be porous with high crosslink network on the hydrogels. The thermal stability of the hydrogels was also determined to range from 68 to 75 °C. The morphology and thermal stability of the synthesized hydrogels determined were relatively of high quality. Therefore, *B. aethiopum* starch could be used in the synthesis of hydrogel via graft copolymerization.

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