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Performance Evaluation of Local Cassava Starches with Imported Starch for Drilling Fluid

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ABSTRACT: *Local cassava starches and an imported starch were characterized and evaluated for their performance as drilling fluid additives. Two local cassava cultivars, TMS 98/0581 and TMS 96/1632, starches and an imported starch were characterized by using X-ray diffraction techniques. Bentonite mud beneficiated with the starches were subjected to API viscosity and filtration tests. The local and imported starches were classified as B type crystal structure. The starches average particle sizes were 51.63nm, 23.57nm and 57.94nm for TMS 98/0581, TMS 96/1632 and imported starches respectively. The crystalline indices were between 22 and 35, while average specific surface areas were 151.65, 361.28 and 142.04 m^2/g for TMS 98/0581, TMS 96/1632 and imported starches respectively. The morphology indices, ranges were from 0.5 to 0.7485. The structural properties of TMS 98/0581 starch were found to be significantly close to that of the imported starch and were observed to be closer in performance as drilling fluid additive than TMS 96/1632. Local cassava starches with structural properties as TMS 98/0581, could serve as good substitute for drilling operation. The viscosity and fluid loss control results of the local samples compared favourably with the imported one.*
Keywords: *X-ray diffraction, drilling fluid, starch, local cassava, crystal structure, bentonite*

I. INTRODUCTION

Polymer additives have been some of the earliest and most common additives for drilling mud. Based on their structure and reactive groups, polymers are used as viscosifiers, thinners, flocculants, surfactant and fluid loss control agents. Starch was the first [1] and the most widely used polymer in drilling fluid. Starch is mainly used as effective colloids, which decreases the filtration of water dispersing drilling fluids and increases the viscosity. The water-swellable amylose enables starch to exhibit fluid loss control properties. Cassava starch, a polysaccharide polymer, has been investigated to exhibit different properties that have potential for a wide end use.

Cassava (*Manihot esculenta Crantz*) is a root crop cultivated in many regions of the developing world. World production of cassava root was estimated to be 252 million tonnes in 2011 [2]. Nigeria is the world's largest producer of cassava with annual production of 45 million metric tonnes in 2008 [3]. Various native and improved varieties of cassava with different physicochemical properties have been developed in Nigeria [4]. Similarities were observed in the physiochemical properties of cassava starch from TMS 98/0505 and polyanionic cellulose (PAC), a commercial drilling fluid additive for viscosity enhancement and fluid loss control [5]. Starch properties from local cassava cultivars were found to influence the performance of water based mud [6]. Enhanced performance was reported for mud formulated with imported carboxyl methyl cellulose (CMC) when beneficiated with local cassava starch [7]. The granule size of cassava starch was reported to affect some functional properties of cassava, such as swelling, solubility and digestibility [8]. The starch granule was described as semi-crystalline [9].

Starch is a morphologically complex polymer substance which consists of loose amorphous regions that are interspersed with regular crystalline regions, resulting from the formation of hydrogen bonds between the starch molecules [10]. X-ray diffraction (XRD) techniques have been employed to investigate the degree of uniformity of molecules by identifying the crystal structure and regular molecular arrangement [11]. XRD pattern in polymer was described as the "finger print" of the crystal structure within starch grains [12]. The crystalline parts give sharp narrow diffraction peaks and the amorphous part gives a very broad peak.

Crystallinity is an indication of amount of crystalline region with respect to amorphous content in polymer. The differences in crystallinity values of starches may also be ascribed to different content of amylose (Fig. 1) and amylopectin (Fig. 2). Starch with lower degree of crystallinity, the structure of amylopectin may be more amorphous. Starches tend to present suitable crystalline arrangements based on their botanical origin.

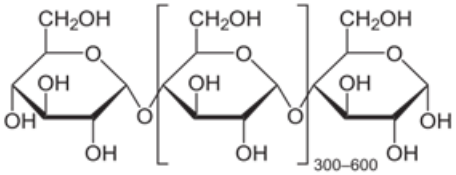

Figure 1: Structure of amylose

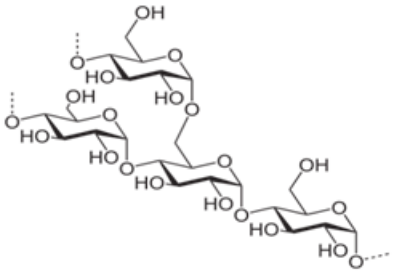

Figure 2: Structure of amylopectin

The crystal structure of starch can be divided into four types, A, B, C and V type [12]. While A, B and C are the crystal structures of natural starches, the V type are complexes formed by amylose and lipids. A-form starch gives stronger diffraction at 15^o, 17^o, 18^o and 23^o. The absence of peak in the range, 5^o to 6^o is a characteristics of an A type starch. B-form has its strongest diffraction at 17^o and other smaller peaks at 20^o, 22^o and 24^o [13]. C pattern is a mixture of A and B [14]. A-form is mainly present in maize, rice and wheat starch. The B is usually available in tuber starch, such as potato. The tuberous starch patterns are recognized by their intensity at one small peak at 5.5, doublet at 17 and a doublet at 23 and 24 [15]. C type diffraction pattern was reported by Uthumporn *et al.* [16] in sago starch samples with peaks at 5.5^o, 10^o, 11.1^o, 15^o, 17.2^o, 17.9^o, 23.5^o and 26.5^o 2θ angles. 17.2^o is a characteristic of B pattern and 23.5^o is a characteristic of A pattern. Nuwamanya *et al.* [17] classified cassava starch as A type. They studied five varieties of cassava and reported their crystallinity between 28.89 and 40.36 percent when their amylose content ranged between 17 and 20 percent. Valencia *et al.*[18] reported that cassava starch showed a diffraction pattern of an A type crystal, characterized by diffraction peaks at angle of $2\theta = 15^o$, 17^o, 18^o and 23^o. In Sandoval and Fernandez [19] studies, cassava spectrum characterized a crystalline A type with peaks at 15.3^o, 17.3^o, 18^o, 23.3^o with crystallinity values at 20 to 23 percent. Cassava starch was also reported to indicate prominent peaks at 2θ positions at 16.44^o, 18.02^o, 35.19^o, 39.23^o and 41.51^o with degree of crystallinity at 20.6 percent using the method of Segal *et al.* [20]. On the contrary, Costa *et al.* [15] reported B type X-ray diffraction pattern for native cassava starch, with doublet at 17^o and 18^o and a single peak at 23^o. The purpose of this work is to characterize and compare an imported starch and two local cassava starches using X-ray diffraction techniques and evaluate their viscosity and filtration performances in drilling fluid.

II. MATERIALS AND METHODS

Local cassava cultivars, TMS 98/0581 and TMS 96/1632 were obtained from National Root Crop Research Institute (NRCRI), Umudike, Nigeria. The twelve months old cultivars were processed to starches by method described by Eke *et al.* [21]. The imported starch was obtained from POCEMA limited, Nigeria. The local cassava and imported samples were used to formulate drilling mud and tested. The samples were also subjected to X-ray diffraction analysis.

2.1 Mud Preparation and Tests

Each of the starch was used to modify bentonite muds according to API 13A specification, 2010. 22.5g of bentonite with 0.5 percent starch (dry weight of bentonite) was weighed with electronic weighing balance, 'A and D' -model FX-5000i, and added to 350ml of distilled water in a beaker. Hamilton Beach mixer was used to stir the suspension for a total of 20 minutes. At every 5 minutes the beaker was removed from the mixer and the sides were scrapped with spatula to dislodge bentonite/polymer clinging to the walls of the beaker.

The bentonite-polymer suspensions were poured into the viscometer cup and subjected to shear in model 800 OFITE 8-speed viscometer. The shear was done at 600, 300, 200, 100, 60, 30, 6, and 3 rpm respectively. At each speed the dial reading was taken when the speed of rotation was stabilized. The rheology test was conducted for each sample at 80^oF, 120^oF, 150^oF and 190^oF. Filtration test for mud samples were performed at 80^oF using OFITE filtration test equipment.

2.2 X-ray Diffraction Analysis and Calculations

2.2.1 X-ray Diffraction Analysis

The polymers were subjected to X-ray diffractometer and data was taken for 2θ range of 0^o to 70^o. They were analyzed in the machine for mineralogy, inter-planar spacing, relative intensity and full width at half maximum (FWHM). The analyses were governed by Bragg's law [20]:

$$2d\sin\theta = \lambda n \quad (1)$$

Where;
n = number of sample introduced in the machine per unit time (usually 1)
λ = wavelength
d = inter-planar spacing or d-spacing and
θ = angle of reflection

2.2.2 X-ray Particle Size Calculation

Considering the peaks at degrees, average particle sizes were estimated by using Debye-Sherrer formula [14]:

$$D_p = \frac{0.9\lambda}{\beta cos\theta} \quad (2)$$

Where,
λ = wavelength of x-ray (0.1541 nm)
β = FWHM (full width at half maximum) in radians
θ = diffraction angle in radians
D_p = particle diameter
0.9 represents Sherrer's constant
θ and β in degree were converted to radians by multiplying with the factor 0.01745

2.2.3 X-ray Degree of Crystallinity

Starch is a semi-crystalline polymer. Amylose forms the crystalline region, while amylopectin forms more of the amorphous region. Relative crystallinity of starch is the percentage of crystal material in total starch [22]. The degree of crystallinity could be calculated, using software [11] by the equation:

$$C_{rl} = 100\left(\frac{A_c}{A_c+A_a}\right) \quad (3)$$

Where A_c and A_a are the area of crystallinity region and area of amorphous region of a sample respectively. However, crystallinity in this work was calculated by empirical method of Segal *et al.*as described by Omotoso *et al.* [20]:

$$C_{rl} = 100\left(\frac{I_{max}-I_{am}}{I_{max}}\right) \quad (4)$$

Where
C_{rl} = the degree of crystallinity
I_{max} = the maximum intensity of lattice diffraction
I_{am} = the intensity of diffraction in amorphous region

2.2.4 Specific Surface Area

Specific surface area (SSA) is value that is employed to determine the type and properties of the material [23]. The SSA of starch could significantly impact on its adsorption and reactions properties.
Specific surface area is given by the equations:

$$SSA = \frac{SA_{part}}{V_{part}*density} \quad (5)$$

or

$$S = \frac{6*10^3}{D_p \rho} \quad (6)$$

Where SSA and S are specific surface area, SA_{part} is surface area of particle,V_{part} is volume of particle, D_p is size of particle diameter and ρ is density of particle. According to Tanetrungroj and Prachayawarakorn [11], equations (5) and (6) would produce the same results.

2.2.5 Morphology Index

A XRD morphology index (MI) is developed from FWHM of XRD data to understand the relationship between the structure and size of the material [14]. MI relates the FWHM of two peaks to its particle morphology (peak having highest FWHM) and a particular peak's FWHM for which MI is calculated). MI was calculated with the following equation:

$$MI = \frac{FWHMh}{FWHMh+FWHMp} \quad (7)$$

Where MI is morphology index, FWHMh is highest FWHM value obtained from peaks and FWHMp is the value of particular peak's FWHM for which MI is calculated.

2.3 Statistical Analysis

The IBM SPSS 23 was employed for statistical correlations.

III. RESULTS AND DISCUSSION

3.1 Crystal Structure Classifications

The crystal structures of the starches were classified according to peaks found in them in the XRD results (Figs.3, 4 and 5). TMS 98/0581 starch was classified as B type. It had peaks at 3.25°, 8.96°, 9.12°, 17.23°, 18.24°, 24.11°, 26.62°in the 2θ positions. It had peaks (doublet) at 17.23° and 18.24° and a peak at 24.11°, similar to the views of Costa *et al.* [15]. It had no peak at 15° 2θ positions, which is a characteristic of A type. Zeng *et al.* [12] described a typical type A X-ray diffraction pattern at 2θ with the first peak at 15°, the second peak near 18° and the third main reflection around 23°. Local cassava, TMS 96/1632 starch was also classified as B type. It has 2θ peaks positions at 3.16°, 3.58°, 17.20°, 18.05°, 24.05° and 28.23°. Imported starch has 2θ peak positions at 6.19°, 19.35°, 24°, 24.92°, 25.18° and 25.36°. It could be described as a B type starch crystal structure. The peak at 6.19° is a feature of B type starch structure. B type structure is a characteristic of tuberous starch [24].

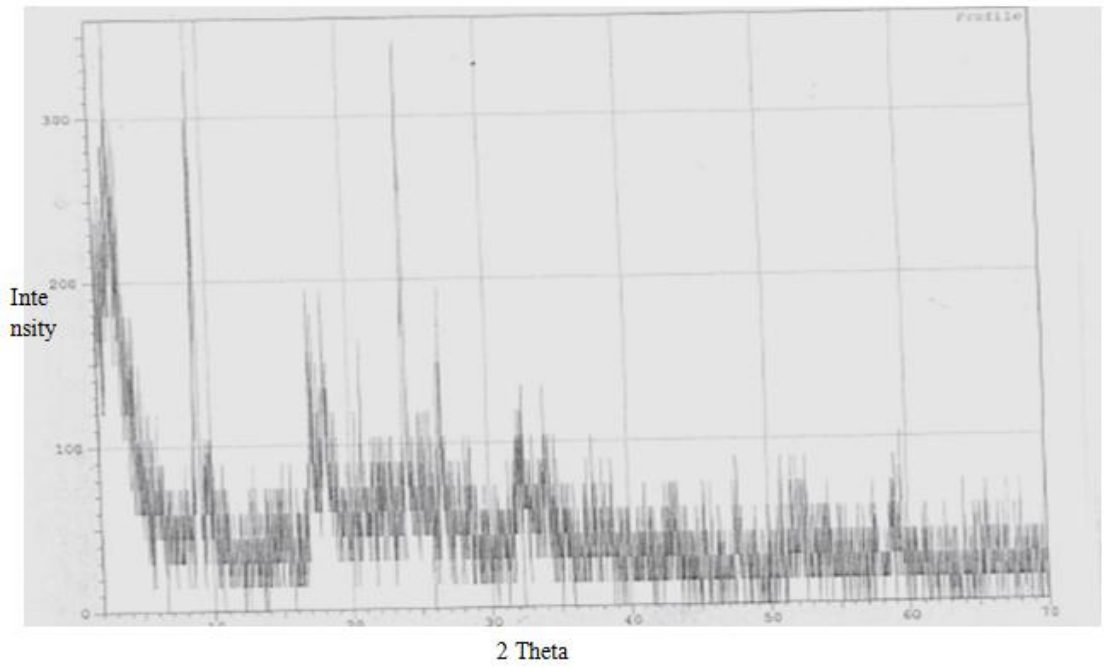

Figure 3: XRD pattern of local cassava TMS 98/0581starch

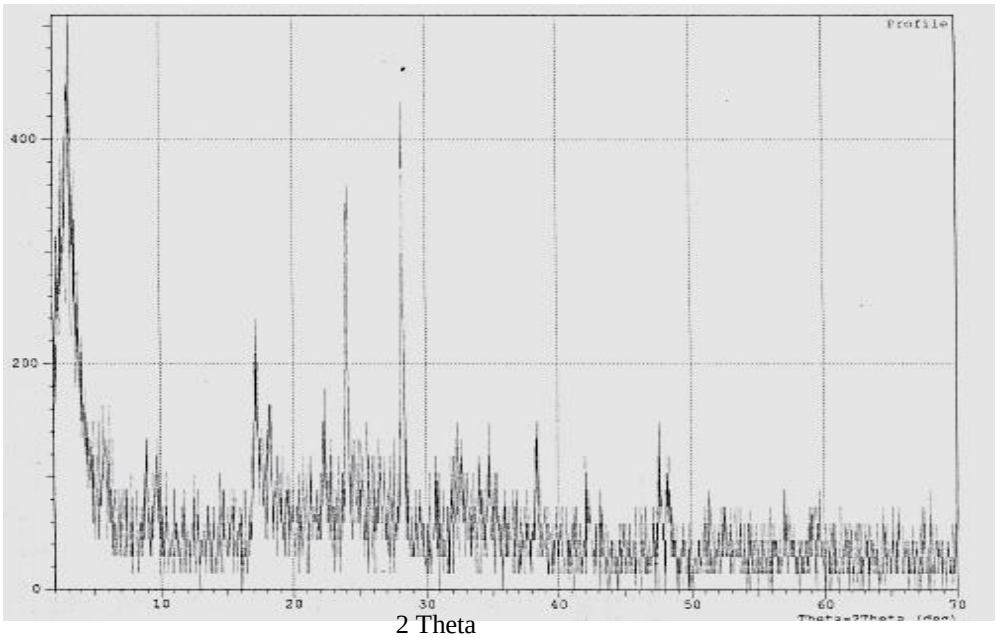

Figure 4: XRD pattern of local cassava TMS 96/1632 starch

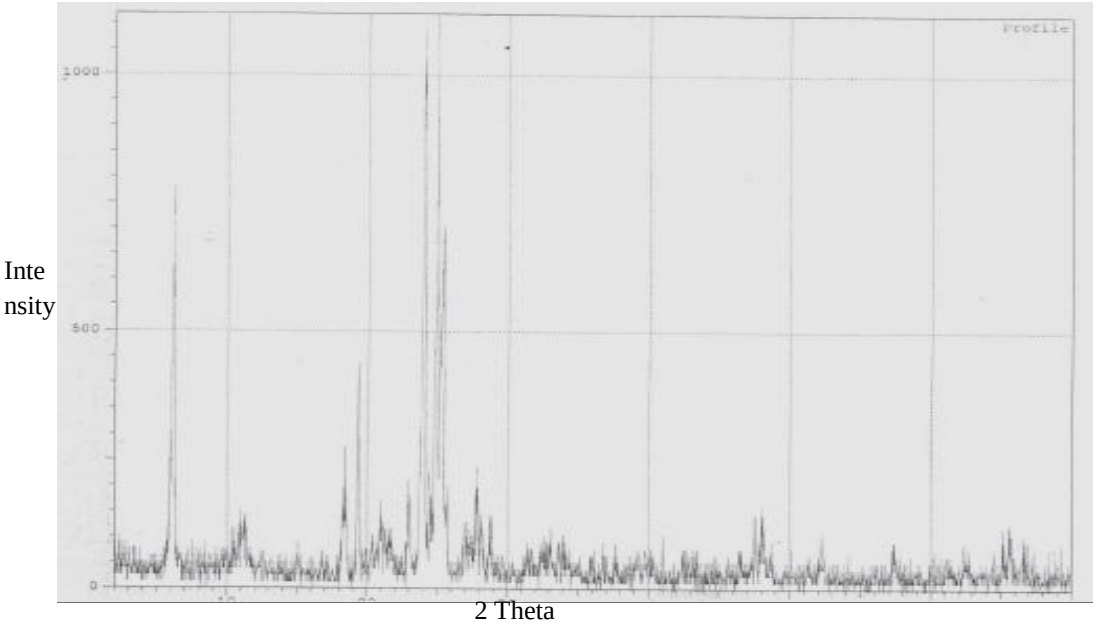

Figure 5: XRD pattern of imported starch

3.2 Mineral Composition

The crystallographic analysis indicated the presence of chemical compounds. The most prevalent compounds for TMS 98/0581 starch are magnesium hydroxide, potassium aluminum silicate hydroxide, calcium oxide, calcium sulfate, sodium aluminium silicate and titanium oxide. For TMS 96/1632 starch, the most prevalent chemical compounds are titanium nitride, magnesium aluminium silicate hydroxide, calcium silicate, potassium aluminum silicate hydroxide, magnesium silicate hydroxide and potassium sodium aluminum silicate. The most prevalent chemical compounds for the imported starch are magnesium oxide, zinc oxide, aluminium oxide, magnesium hydroxide, titanium oxide and magnesium aluminium silicate hydroxide.

3.3 Particle Size of Starches

The particle sizes of starches as calculated from equation (2) are contained in Tables 1, 2 and 3. The particle sizes for TMS 98/0581 starch ranged from 35.01 to 71.18nm. Particle size of TMS 96/1632 starch ranged from 12.42 to 37.80nm. Particle size for imported starch ranged from 38.38 to 76.97nm. TMS 98/0581 and imported starch had close particle size limits. The average particle sizes for TMS98/0581 starch and imported starch were 51.63nm and 57.94nm respectively, while that of TMS96/1632 starch was 23.57nm. Particle size of native cassava starch reported by Omotoso *et al.*[20] ranged between 50.05 to 50.66nm.

Table 1: The particle size of TMS98/0581 starch

2θ (deg)	FWHM of peak (β) (radians)	Size of the particle (nm)	d-spacing (nm)
3.2500	0.00244	56.79	27.16360
8.9600	0.00195	71.18	9.86159
9.1200	0.00303	45.98	9.68894
17.2316	0.00273	51.30	5.14152
18.2400	0.00314	44.72	4.85985
24.1120	0.00251	56.44	3.68798
26.6233	0.00407	35.01	3.34553

Table 2: The particle size of TMS 96/1632 starch

2θ (deg)	FWHM of peak (β) (radians)	Size of the particle (nm)	d-spacing (nm)
3.1600	0.01117	12.42	27.93705
3.5800	0.00663	20.93	24.66048
17.2050	0.00750	18.69	5.14980
18.2150	0.00681	20.64	4.86647
24.0475	0.00375	37.80	3.69773
28.2275	0.00462	30.93	3.15894

Table 3: The particle size of imported starch

2θ (deg)	FWHM of peak (β) (radians)	Size of the particle (nm)	d-spacing (nm)
6.1890	0.00341	40.76	14.26932
19.3533	0.00186	75.56	4.58273
23.9965	0.00369	38.38	3.705747
24.9211	0.00196	72.54	3.57005
25.1800	0.00185	76.97	3.53393
25.3629	0.00327	43.42	3.50885

3.4 Crystallinity of Starches

The relative intensities of the outstanding peaks are in Tables 4, 5 and 6. For TMS98/0581 starch, the I_{max} was found at 2θ peak, 24.1120^o with relative intensity of 100 percent and I_{am} was found at 2θ peak, 8.9600^o with relative intensity of 65 percent. For TMS96/1632 starch, the I_{max} was found at 2θ peak, 28.2275^o with relative intensity of 100 percent and I_{am} was found at 2θ peak, 3.1600^o with relative intensity of 78percent. I_{max} for imported starch was found at 2θ peak, 23.9965^o with relative intensity of 100 percent and I_{am} was found at 2θ peak, 25.3628^o with relative intensity of 65 percent. The degrees of crystallinity of starches as calculated by empirical method of Segal *et al.* (equation 4) were 35, 22 and 35 percent for TMS 98/0581, TMS 96/1632 and imported starches (Fig. 6) respectively. Degree of crystalinity values ranging from 20 to 40 have been reported for cassava starch [19],[17], [25], [24]. TMS 98/0581 and imported starches had same values in their degrees of crystallinity.

Table 4: Intensity of XRD peaks for TMS98/0581 starch

2θ of peak (deg)	3.2500	8.9600	9.1200	17.2316	18.2400	24.1120	26.6233
Relative intensity	43	65	96	35	35	100	35

Table 5: Intensity of XRD peaks for TMS 96/1362 starch

2θ of peak (deg)	3.1600	3.5800	17.2050	18.2150	24.0475	28.2275
Relative intensity	78	52	52	37	78	100

Table 6: Intensity of XRD peaks for imported starch

2θ of peak (deg)	6.1890	19.3533	23.9965	24.9211	25.1800	25.3629
Relative intensity	70	42	100	96	52	65

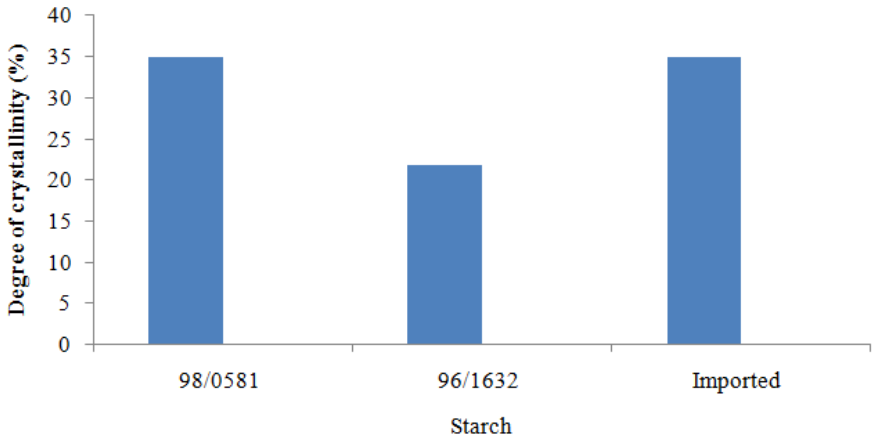

Figure 6: Degree of crystallinity of starches

3.5 Specific Surface Area of Starches

The specific surface area (SSA) of TMS 98/0581, TMS 96/1632 and imported starches (Tables 7, 8 and 9) were evaluated. SSA values for local cassava starch TMS 98/0581 ranged from 105.37 to 214.24m^2/g. TMS 96/1632 starch SSA ranged from 198.44 to 603.71m^2/g, while that of imported starch ranged from 97.44 to 195.41m^2/g. Average values of SSA were 151.65, 361.28 and 142.04 m^2/g for TMS 98/0581, TMS 96/1632 and imported starches respectively. SSA range of 148.05 to 149.85m^2/g for native cassava starch was reported [20]. The SSA of imported starch was not significantly different from SSA of TMS 98/0581starch ($p > 0.05$), but significantly different from SSA of TMS 96/1632 starch ($p < 0.05$). Specific surface area of starch particles is inversely proportional to the particle sizes.

Table 7: Specific surface area of TMS98/0581 starch

FWHM of peak (B) radians	Size of the particle (Dp) nm	specific surface area (m^2/g)
0.00244	56.79	132.06
0.00195	71.18	105.37
0.00303	45.98	163.11
0.00273	51.30	146.20
0.00314	44.72	167.71
0.00251	56.44	132.89
0.00407	35.01	214.24

Table 8: Specific surface area of TMS96/1632 starch

FWHM of peak (B) radians	Size of the particle (Dp) nm	specific surface area (m^2/g)
0.01117	12.42	603.71
0.00663	20.93	358.41
0.00750	18.69	401.21
0.00681	20.64	363.39
0.00375	37.80	198.44
0.00462	30.93	242.52

Table 9: Specific surface area of imported starch

FWHM of peak (B) radians	Size of the particle (Dp) nm	specific surface area (m2/g)
0.00341	40.76	184.03
0.00186	75.56	99.26
0.00369	38.38	195.41
0.00196	72.54	103.38
0.00185	76.97	97.44
0.00327	43.42	172.71

3.6 Morphology Index

The morphology indices (MI) were calculated from equation (7).The morphology index of local cassava TMS 98/0581 starch ranged from 0.5 to 0.6756, which had close value ranges with that of imported

starch at 0.5 to 0.6667. The morphology index of local cassava TMS 96/1632 starch ranged from 0.5 to 0.7485. In all cases, 0.5 MI value was the lowest from the provision of the formula. Morphology indices have direct relationship with particle sizes and inverse relationship with specific surface areas of the starches (Figs. 7 and 8). The morphology index had good correlation with particle size as evaluated forTMS98/0581 starch (R^2=0.9868), TMS96/1632 starch (R^2=0.9748) and imported starch (R^2=0.9988). It was observed that local cassava starch TMS 98/0581 has closer morphology index versus particle size relationship with the imported starch. The local starch TMS96/1632 correlation deviates at 2.4 percent from imported starch and 1.2 percent from TMS98/0581. Morphology index versus specific surface area of the starches showed closeness between TMS98/0581 and imported starch.

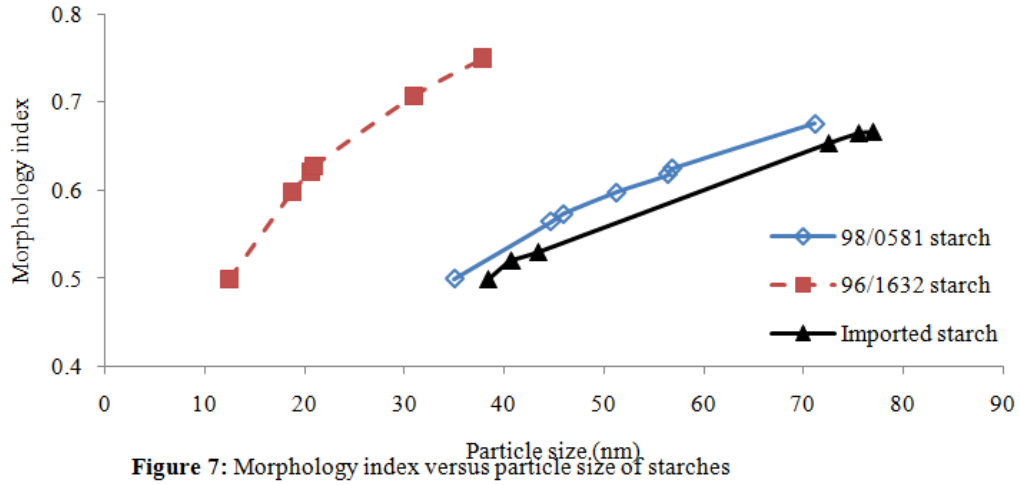

Figure 7: Morphology index versus particle size of starches

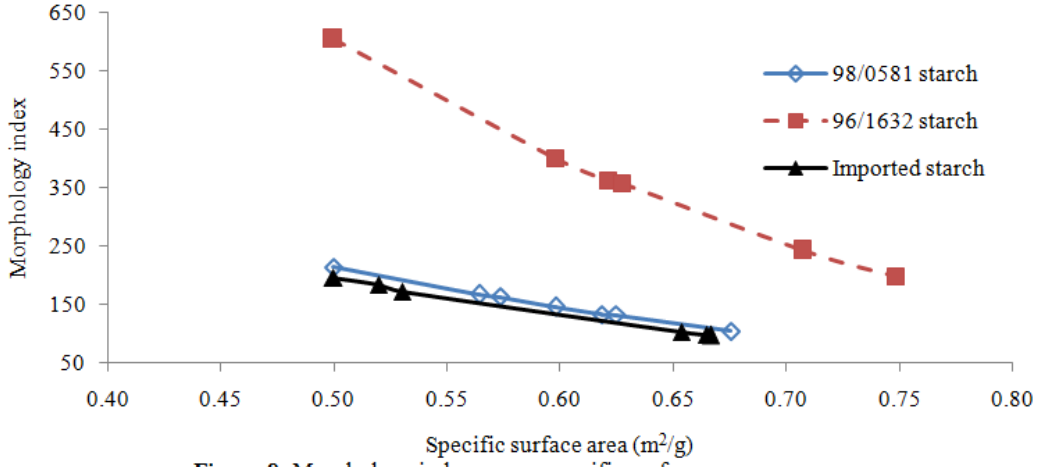

Figure 8: Morphology index versus specific surface area

3.7 Effect of Starch Structure on Mud Performance

The viscosity of bentonite-polymer mud is depicted in Fig. 9. At 600rpm the viscosities of the mud formulated with imported starch ranged from 39 to 48cp between temperatures of 80°F and 190°F. The viscosities of bentonite –TMS 98/0581 starch mud ranged from 36 to 43cp, while that of TMS 96/1632 starch ranged from 31 to 34cp. For a bentonite mud, the API minimum is 30cp [26]. The imported starch, at 0.5 percent concentration in bentonite mud had the best performance. TMS 98/0581 starch was close to the imported starch in performance as a viscosifier, than TMS 96/1632 starch.

The starches reduced the fluid loss in bentonite muds (Fig. 10). The fluid loss for bentonite mud without polymer additive was 18cp. The imported starch reduced bentonite mud fluid loss by 30 percent. TMS 98/0581 starch reduced bentonite mud fluid loss by 20 percent, while TMS 96/1632 starch reduced fluid loss by 14.44 percent.

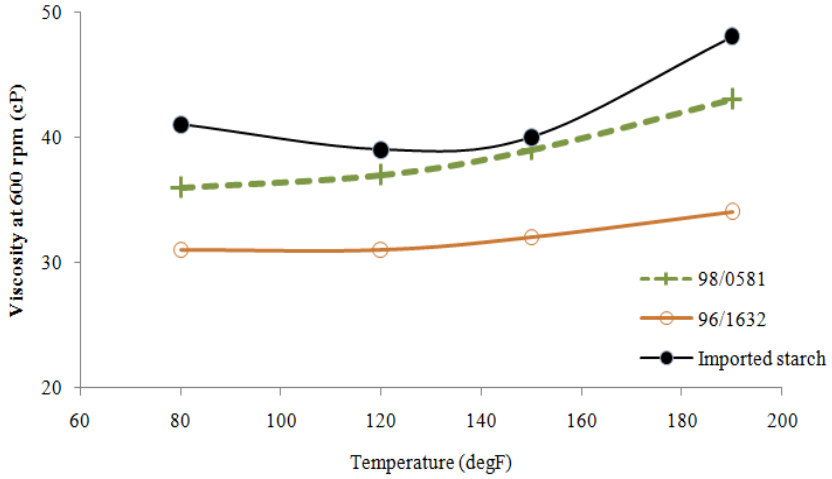

Figure 9: Viscosity bentonite-polymer mud at 600 rpm

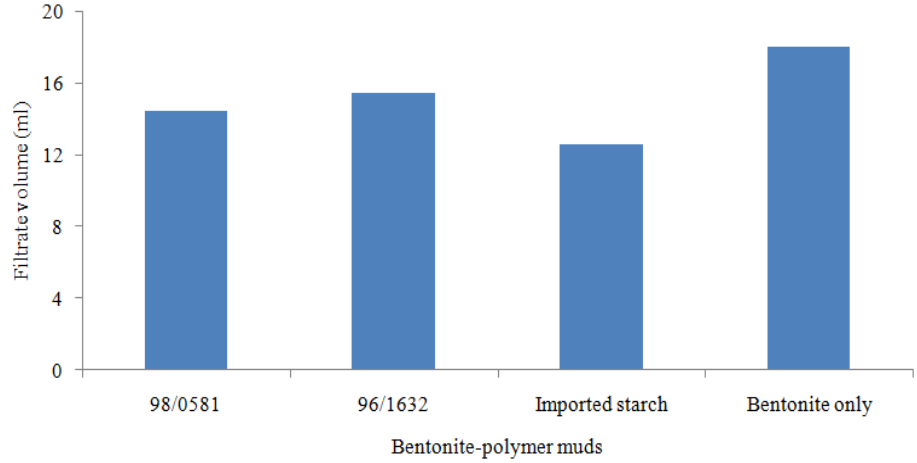

Figure 10: API fluid losses of bentonit-polymer muds

IV. CONCLUSION

X-ray diffraction techniques were used to characterize two local cassava starches from TMS 98/0581 and TMS 96/1632 and an imported starch. Bentonite muds were formulated with the respective starches and rheological and filtration tests were performed. The bentonite mud formulated with TMS 98/0581 was next in performance to the best performed bentonite mud sample formulated from the imported starch. TMS 96/1632 starch formulated bentonite mud performed significantly lower than the others. The mineralogy, crystalline index, particle size, specific surface area and morphology index values of the two best performers were found to be close. High particle size and crystalline index of starch favour the performance of bentonite mud. Imported starch could be substituted with local cassava TMS 98/0581 starch as drilling fluid additive. The structural properties of starches contributed to their behaviours in drilling fluid performance

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