Evaluation of Physico-Chemical Properties of starches from *DisocoreaRotundata*species

Umaru Ahmadu[a], Yusuf A.S.[a] and Oluwatoyin Odeku[b]

[a]Department of Physics, Federal University of Technology, Minna, Nigeria

[b]Department of Pharmaceutics and Pharmacy , University of Ibadan, Nigeria

Corresponding author:u.ahmadu@yahoo.com

Abstract

Three species of starches from *dioscorearotundata:Giwa, Lagos, Sule and Kwasi* were investigated and characterized by X-ray diffraction, EDX/XRF/PIXE/SEM with Proximate composition determined.Yams tarches were found in all the *D.R.* species.XRD peaks of highest intensity are at~17.02,17.12 and 17.26°2θ with relative crystallinities (%) ~ 18.25$\pm$1.25, 22.25$\pm$5.75, and 18.75$\pm$0.75%, respectively.A distinctive characteristic is that all the species contain elements Ru(18.37 to 27.71) and K (11.48 to 21 w/w%)in major concentrations. Their microstructure has grain sizes of 3.59, 3.54 and 3.19 μm, respectively, for*Giwa, Lagos*and*Sule.* The grains are generally uniform/oval in shape and mixtures of B and C-crystal types.The crystallite sizes are ~2.5, 3.0 and 2.4 nm, for *Giwa*, *Lagos*and*Sule,* respectively.Whole profile fitting of the patterns with subsequent refinement using GSAS II suite of programs, together with instrumental resolution curve fitting and peak profile analysis show agreement factors (*Rwp)* of 1.49, 2.66 and 1.75%, and were all indexed as orthorhombic cells.It is concluded that the starches can be stored safely due to their low moisture content, the possibility of some mineral deposits in the soil and that they have physico-chemical properties that may be suitable for application in the starch industry, particularly when modified.

A. Introduction

Yam tuber is one of the most staple foods eaten in sub-Saharan Africa and constitutesabout 15% of

total calories and 81% of protein of the average Nigerian diet(Odebami*et al*.,2007), compared to the

UK , for example,where it is about 30% of diet by weight(Wang *et al*., 1998). White yam (guinea

yam or African yam) belongs to the *DioscoreaRotundata* (*D.R.*) family with over 600 species in which only seven are edible (Polycarp *et al.*, 2012; Amani *et al.*, 2005) or cultivated for food and medicine. According to the International Institute of Tropical Agriculture(IITA), in 2005 yams were produced in 47 countries (Polycarp *et al.*, 2012) in tropical and subtropical regions of the world and out of the 47.8 million tons produced, 97% were from sub-Saharan Africa, Nigeria being the leading producer with 34 million tons.

Dioscorea species contain carbohydrates which essentially are starches of different origins with varying degrees of crystallinity of between 15 -45%(Napaporn*et al.*, 2001) and can be extracted from various starch bearing tubers, roots or cereals. Inspite of the predominance and relevance of this species there is dearth of literature in the area of work carried out on yam starch and particularly, on exploiting their favourable physical properties for industrial applications with major data bases reporting less than 1%(Amani *et al.*,2005; Satin, 2006) compared to other sources such as potato, corn,maize and rice.Further, there is little attempt at correlating the various properties of the *dioscorearotundata*species found within and outside Africa with different local names with the view to classifying them based on their similar properties

Native starches are composed of amylose and amylopectin which form the amorphous and crystalline components, respectively(Napaporn*et al.*,2001; Karin *et al.*, 2003) and their crystallinity have been classified as A, B or C based on Wide Angle X-ray Diffraction(WAXD)(Riley *et al.*, 2006;Frost *et al.*, 2009). Detailed structure of starch and other relevant features have been described by Karin *et al.*(2003) and Whaig*et al.*,1997). Starch has been studied by various techniques to elucidate some of its physical properties, such as *in situ* SAXD(Small Angle X-ray Diffraction), WAXD(Jenkins and Donald, 1997), SEM, NMR, AFM and TEM(Cornnejols and Perèz,2010) and impedance spectroscopy, among others (WawroKazimierczak, 2008; German*et al.*, 2012).

The above underscores the relevance, challenges and necessity to understand the physical, chemical and material properties of starch biopolymers from *D.R.* sources in sub-saharan Africa. It also underlines the importance of correlation studies within and outside Africa of the properties of *D.R.* species. In this work the results from proximate composition, material properties, mineral composition, microstructure and X-ray diffraction studies of starch have been analysed fort hree local varieties of*D.R.*, namely, *Giwa (G), Lagos (L) and Sule (S)*). The results have been correlated with similar *D.R.* species' properties and evaluated for suitability in relevant starch industries.

B. Experimental

1. Sample.

Three species of*D. R.*, i.e., G, L and S were harvested from Pmazi village in Bosso,Minna, Niger state, Nigeria. They were harvested fresh from the farm and a month later extraction of starch was carried out.

2. Specimen preparation

Each tuber of the yam cultivars was weighed in a top loading balance before being peeled and reweighed. This was followed by cutting in pieces and blending with 100ml of distilled water using electrical blender. It was followed by sieving with five litres of distilled water which was allowed to settle for about ten minutes and decanted. Decantation was repeated three times. The starch collected was then spread on a clean plastic tray and allowed to dry at room temperature for about 24h. The dry starch was reweighed and the percentage starch contained in each tuber was determined.

3. Data Collection

Three specimens of each sample were subjected to X-ray diffraction to ensure consistency and repeatability of results. The measurements were carried out with a Philips X'pert pro working at 30

mA, 40 kVin a continuous scanning mode in reflection-transmission mode at 0.0670^o 2Θ step size in the 2Θ range 5.07 to 8.96^o with Cu $k_{\alpha 1} = 1.5405\ A^o$ at 25^oC and Cu $k_{\alpha 2} = 1.54443\ A^o$.

Specimen starch powders were gold-sputtered and analysed in a scanning electron microscope, ESEM 30, Philips, Kassel, Germany at an accelerating voltage of 2 kV.Particle size distributions were determined by laser light diffractometry using a dry feeder (Malvern 2600C, Malvern Instruments, Worcestershire, U.K.). The feeder was set at a pressure of 400 kPaand the injector to a pressure of 6 kPa.The focal distance was 300 mm and the measuring time was 25-35 s. The mean particle size was determined in quadruplicate.

The apparent particle densities of specimens of all equilibrated starches were determined by helium pcnometry(Acupye 1330,micrometrics, Norcross, GA, USA) in triplicates (Picker and Mielck(1996).

Bulk and tap densities were determined in a 250 ml cylinder suing a volumeter(stampfvolumeterStav 2003, J.Engelsmann AG, Ludwigshafen, Germany). Determinations were also made in triplicate according to the European pharmacopoeia (EP, 2007).

Proximate composition analysis for ash and lipids contents was carried out according to*Association of Official Analysis Chemists* AOAC(2000)methods. Protein content was estimated from the nitrogen content determined by elemental analyses based on a conversion factor of 1.25(Gebre-Mariam and Schmidt, 1998). The phosphorus content was determined from the starch ash by mixing the starch with 1%(w/w) sodium carbonate and ignited in a furnace at 550^oC for 6h.The phosphorus content in the starch was determined colorimetically based on the method described by Murphy and Riley(1962).The amylose content was determined colorimetically using the method described b**y** William *et al*.(1970).All determinations were done in triplicate and the results were presented as mean and standard deviations.

Powder flowability:The flowability of the starches wasassessed using the Hausner ratio and the Carr index (Carr, 1965).The Carr index was calculated as followings:

Carr index =Compressibility = $\dfrac{\text{tap density - bulk density}}{\text{tap density}}$ x 100

The flow rate of the starch powders were determinedusing a steel funnel on a Pharmatest flow rate apparatus (Sartorius Pharmatest, Apparatebau GmbH, Hainburg, Germany) with an orifice of 15 mm. All determinationswere performed in triplicate.

Swelling power and water binding capacity: Swelling power and solubility: The swelling power at room temperature ($27°C \pm 2$ °C) was determined using the method described by Leach *et al*. (1959), whilethe water binding capacity was determined using the method of Ring (1985). All determinations were done in triplicates.

Similarly, elemental analysis and microstructure were carried out on the samples using Rigaku Supermini WD-XRF, 50 kV /200 W using three techniques for complementarity and validation purposes to cover trace concentrations, i.e., SEM-EDX (Carl Zweiss EVO 40 EP, 30 kV)/XRF/PIXE.

C. Results and Discussion

1. Proximate composition of *D.R.* starches

Table 1presents the determinedparameters of the starches with sample S having the highest moisture content in the order S>L>G. Their protein/crude fat/total ash/fibreand carbohydrate contents are similar. The values of their amylose contents are also similar with G being slightly higher.

Table 1: The proximate composition of *Dioscorea* starches

Starch	Moisture content (%)	Crude protein (%)	Crude fat (%)	Total ash (%)	Crude fibre (%)	Crude carbohydrate (%)	Amylose content (%)
Lagos	9.67	1.23	0.385	1.06	1.08	86.596	23.06
Giwa	9.58	1.27	0.426	1.03	1.10	86.609	23.16
Sule	9.98	1.36	0.439	1.09	1.13	86.022	22.86

The work of Odeku and Picker-Fryer(2007) on *D.R.* species showed higher amount of moisture (11.96±0.06%) and amylose (28.830±0.65%) contents, compared to the average value obtained for the same parameters in this work. However, the amounts of crude proteins, lipids and ash reported in their work (0.28±0.08%, 0.02±0.01% and 0.015±0.01%), respectively, are much smaller than our results. In the case of phosphorus determined from proximate analysis in their work (0.022±0.001%),it is lower than the value obtained in this work (0.028±0.002%).Work on four *D.R.*species carried out in Ghana(Ransford, 2012) determined moisture, ash, crude protein, crude fat, crude fibre and carbohydrate contents as(w/w%) 7.22 to 7.82, 0.24 to 0.86,~1.31,~ 0.04, 0.1 to 0.15 and 40.13 to 91.06, respectively; amylose and amylopectin were reported as(%) 27.48±0.47 to 31.55±0.47 and 68.45±0.47 to 72.52±0.47, respectively.The low moisture content determined for the species is far below the threshold of 13%(w/w)(Odeku and Picker-Fryer, 2007) which makes them safe for storage.

2. Material Properties

The results of material properties determined for the *D.R.* species are presented in Table 2.

Table 2: Material properties of *Dioscorea* starches

Starch	Particle size (μm)	Particle shape	Apparent particle density (g/cm³)	Bulk density (g/cm³)	Tapped density (g/cm³)	Carr's Index (%)	Angle of repose (°)	Swelling Capacity (ml)	Water binding capacity
Lagos	3.54	Oval- angular	1.59	0.509	0.635	19.84	65.60	1.20	0.57
Giwa	3.59	Oval	1.57	0.599	0.790	24.17	57.00	1.29	0.67
Sule	3.19	Oval- Oblong	1.49	0.506	0.715	28.81	65.90	1.19	1.20

All the species have small particle sizes. Sample G has higher particle size, followed by L, S being the smallest. The particle shapes are generally uniform and oval, while the apparent/bulk/tap densities are all similar. The values of particle sizes reported by Odeku(2012) and Odeku and Picker-Fryer(2007) are much higher($29.85 \pm 0.17 \mu m$, $18.68 \pm 0.85 \mu m$) when compared to those reported in this work. Figure 1(A to C) shows the microstructure of the surfaces of the *D.R.* species at magnification of x 200.On the other hand, the average particle densities reported by Odeku and Picker-Fryer(2007) (1.53 g/cm^3) are almost the same as that of this work. However, the values of bulk and tap densities reported in their work are higher. The Carr's index for *Lagos* species is the lowest and indicates fair flowerbility whereas *Sule*and *Giwa*have poor flowerbility (but higher compressibility) due to their higher values (Anisko and Piroska*et al,* 2014;Odeku and Picker-Fryer, 2007).The poor flowerbility is due to strong force of cohesion between starch particles which is reinforced by the very small particle sizes providing high surface areas that promote interparticle attraction. These parameters are relevant for application as pharmaceutical excipients.

The work of Otebayo*et al.*(2005) on six species of *D.R.*reported average grain sizes in the range 18.4 ± 5.0 to 40.9 ± 3.0 μm. Riley *et al*(2006) on the other hand, reported polygonal and rodlike shapes for the grains with mean diameters of 27.33 μm.The results indicate grain sizes that are much higher

and wider in range, with the shapes generally in conformity with literature on *D.R.* species as reported in the work of Ransford (2012). Ransford (2012), working on four *D.R.* species in Ghana reported mean grain sizes in the range7.10 to 66.99 µm, swelling power and water binding capacities (%) as 1.05 ± 0.04 to 12.48 ± 0.02 and 1.73 ± 0.26 to 1.02 ± 0.25. Our results therefore show grain sizesthat are much smaller and narrower which has implications for industrial applications such as the paper industry. This is also true of the swelling power and water binding capacities which are both larger and wider in range, and larger and narrower in range, respectively.
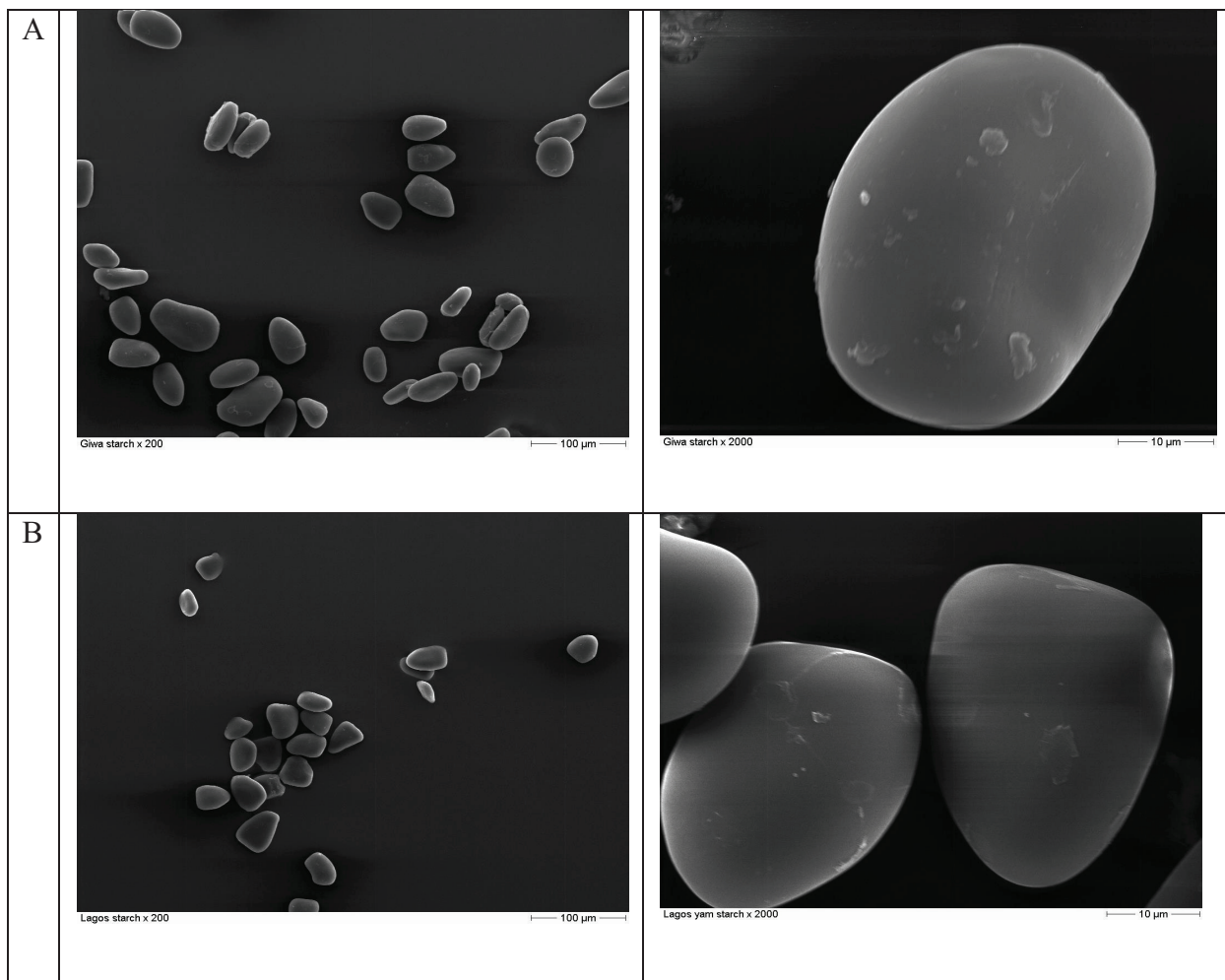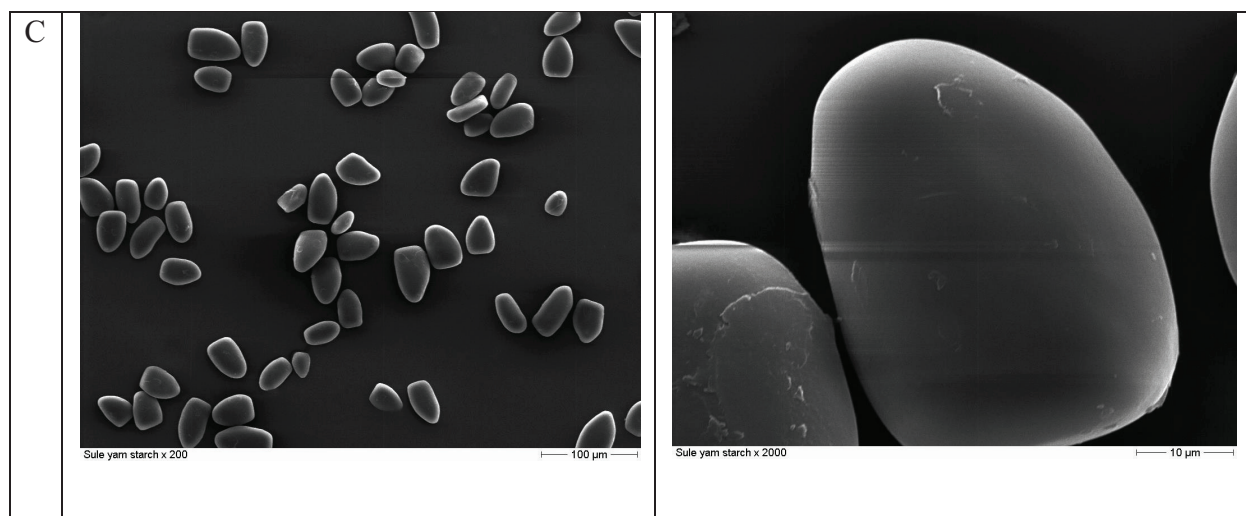
Figure 1. SEM of the different cultivars of yam starches: A,Giwa; B,*Lagos*; C. *Sule*

3. Elemental analysis

Qualitative EDX results ran on samples G, L and S showed the presence of the elements C, K, Al and O, while Cu was observed in sample L in addition. Qualitative XRF resultsalso revealed the presence in sample G of the elements K, Ca, Mn, Zn, Fe, Ru, Cu, Mo, Ni, Br, Sr, As, Cr Se and Co. Samples L and S have similar elemental compositions, except that Ca is absent in both. PIXE results show presence of the elements Mg, Al, Si, P, S, K, Fe, Zn in sample G; Mg and Zn are missing in samples L and S. The most significant results are the quantitative values(w/w%) obtained from XRF with elemental concentrations of K(17.29 ± 2.34) and Ru(26.78 ± 3.75)constituting major concentrations in G;Sample L has K(17.28 ± 2.54), Ru(18.37 ± 3.55) and Mo(10.26 ± 1.39); whereas sample S has K (11.48 ± 2.10) and Ru(27.71 ± 4.30). The presence of Ru has not been reported,but the fact that it is present in major concentrations in all the *D.R* .species make the results not only unique but important owing to the relevance of Ru (as the mineral Rutile(TiO_2)) in the manufacture of optical components.Those present in minor concentrations in sample G are Mo(8.85 ± 1.09); Cu(8.82 ± 0.48) in sample L and Cu(8.08 ± 0.44) in sample S. The concentrations of Cu are almostthe same in all the *D.R.* species. Further, the amount of P determined from PIXE (w/w%) (0.022 ± 0.002-G,

0.035±0.002-L and 0.024±.002-S) and that reported using the method of Murphy and Riley (1962) for *D.R.* species found in south west Nigeria are in good agreement(0.022±0.001) (Odeku and Picker-Fryer(2007). Though the values reported in our present work areslightly higher. Qualitative work reported on mineral compositions of *D.R.* species found in south west Nigeria by Odebumi*et al.*(2007) found the elements Mn, Fe, Zn, Ca and Mg, all of which were detected in our samples. Work of Polycarp *et al.*(2012)on *D.R.* species show the presence of elements (w/w%)K (0.475±0.003), Na (0.070±0.004),Ca (0.100±0.005), Mg (0.035±0.005) and P(0.158±0.017), all less than the values for the present work, particularly, the K. However, the P concentrationreported is much higher than our results and those reported in literature (as mentioned elsewhere).Na was not found in our work.Tubers are associated with high P content(Aprianita, 2010)and haveenormous implications for many physical properties of starch such as viscosity, gelatinization temperature, transparency, among others. These variations have been attributed to geographical and botanical origins, amongst others.

4. X-ray Diffraction

Composite XRD patterns using XPowder program (Martin, 2008) for the three D.R.species are shown in Figure 2 on a normalized relative intensity scale for the species.It is observed that the positions of the central/largest peaks are all coincident and the reflection intensities show good counting statistics compared to those reported in literature. Sample S has the most intense peak amongst them.Table 3 is a summary of the peak(2Θ°) positions, crystallinity (%) and the proposed crystal typesof the *D.R.* species.
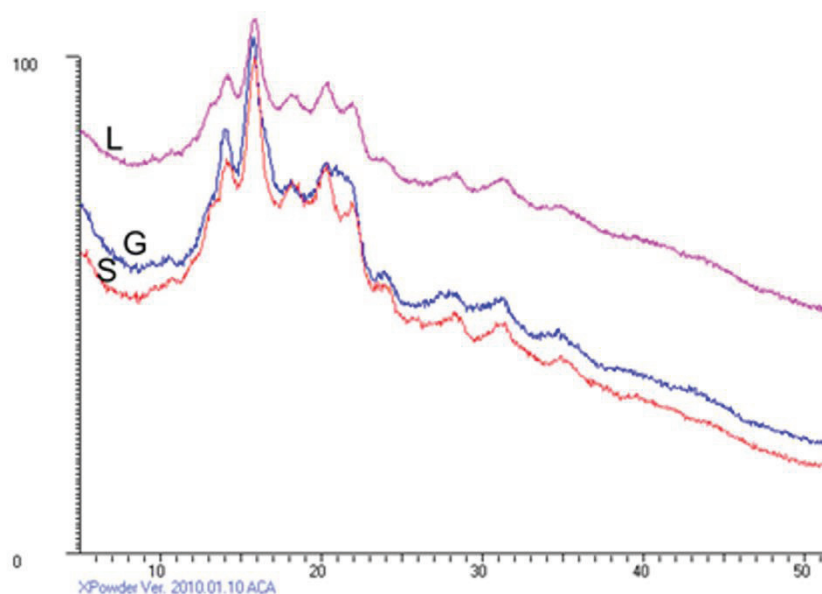
Figure 2.Composite XRD patterns of *Lagos(L)*, *Giwa(G)* and *Sule(S)* on a normalized intensity scale.

Table 3.Summary of peak (2Θ) positions (most intense), crystallinity and crystal type forthree

***D.R.* species.**

Starch species	2Θ(°)	Crystallinity (%)	Crystal type
Sule	5.43,14.03,15.34,17.26,19.82,22.16,24.05	18.75±0.75	B
Giwa	5.27,15.14,17.02,21.96,23.93,34.37	18.25±1.25	B,C
Lagos	5.37,14.01,15.11,17.12,19.91,22.14,23.85	22.25 ±5.75	B,C

There are a number of discernible peaks in the diffractograms of some of the *D.R.* species compared to those reported literature (Riley, 2006; Ao*et al.*, 2007;Yuan *et al.*, 2007;Maria *et al.*, 2010). The strongest peaks occur at 17.02 °,17.12 °and 17.25 ° for samples G, L and S, respectively. Comparison

of the data with those reported at the same wavelength in literature show that they are consistent with reported diffractograms obtained from different starch sources, including *D.R.* species.

Phase composition analysis shows that all the *D.R.* species belong to yam starches. However, based on the reference patterns (ICDD): 00-052-2246(lentil starch), 00-052-2248(yam starch), 00-052-1802(maize starch) and 00-052-2247(potato starch) some of the 2Θ values could be related to these patterns. One of the peaks (17.23°) of sample G for example, corresponds that reported in maize starch (Maria *et al.*,2010) while the peak at 17.2° corresponds to that reported for potato(Yuan *et al.*, 2007) in samples L and S. This is validated by the reference patterns. *D.R.* has been found to exhibit the open hydrated patterns of B-type typical of tuber starches with strong diffraction peaks at $\sim5.8^{\circ}$, a single sharp peak at 17.5° and two identifiable peaks at 22.5° and 24°(Riley, 2000). These figures indicate that the species belong to the *D.R.* family and the crystalline types may be hypothesized to be a mixture of B and C polymorphs (Odeku and Picker-Fryer,2007,2009) because the FT-Raman spectra of *D.R.* are similar to that of maize starch which could further lead to a similarity of molecular structures.XRD patterns of *D.R.* have been shown to vary from B to C-type(Odeku, 2012). Samples L and G may therefore be mixtures of B- and C-crystal types while S may be B-type. The computed relative crystallinities are as presented in the table 3determined from XPowder program(Martin,2008) and show that sample G has the lowest crystallinity of the species.Thesevalues are within the range reported for starches of various origins, 15-45% (Napaporn*et al.*, 2001) and indicate that some of the starches have moderate crystallinity.

The crystallite sizes (D) were analysed by the XPowder program using Debye-Scherer equation ($D = \frac{k\lambda}{10.\beta Cos\theta_{\beta}}$, (nanometers),with k=1; whereβ, λ and θ_{β} are the fullwidth at half maxima(FWHM), wavelength and Bragg peak, respectively).Values (for $2\Theta=16.34^{\circ},16.47^{\circ}$) for sample G are 2.4 and

2.3 nm, respectively; whereas for sample L, we have (2Θ=17.039°,17.172°) crystallite sizes of 2.5 and 2.4 nm, respectively,after$k_{\alpha1}$, broadening correction and neglecting strain. The results indicate that Samples S and G have similar crystallite sizes. The crystallites are very small hence the broadenings observed in the XRD patterns which are attributed to sample size effects only (after instrumental correction).The small size of the crystallites explains the modest crystallinities of the species.Table 4 is a summary of the profile parameters for calculating the crystallite size. Theasymmetry, shape factor and instrumental Cagliotti coefficients for selected 2Θvalues based on simple Gaussian analysis are shown.

Table 4.Profile parameters and Instrumental Cagliotti coefficients

Species	2Θ°	*FWHM(2Θ°)	Crystallite size(nm)	Assymetry	Shape factor	Instrumental Cagliotti Coefficients		
						U	V	W
Giwa	16.34	3.79	2.5	-0.31	0.63	0.014680	0.005190	0.005800
Sule	17.04	3.56	2.4	-0.33	0.61	0.014680	0.005190	0.005800
Lagos	15.87	3.02	3.0	1.50	0.51	0.014680	0.005190	0.005800

*All FWHM are after $k_{\alpha2}$and Instrumental broadening corrections.

5. Full Profile Fitting, Refinement and Indexing

Whole(and single peak) profile fittingsfor samples S, G and L and subsequent refinement were carried out with GSASIIprogram (Von Dreel and Torby, 2010) for peak positions, intensity and background, while the peak widths were fitted using Gaussian and Lorentzian parameters as shown in Figure 4(A–F). Figure 4(A, B) for sample Grepresentssingle peak fit and Gaussian and Lorentzian fits for instrumental functions. The plot shows the resolution curves corresponding to values of the Gaussian (U, V, W), and Lorentzian X, Y coefficients. The '+' marks show the individual values based on sigma and gamma values for the peaks (i.e., deviations).The discontinuities seen in the data points are bad reflections which were omitted during refinement. The green continuous line is the fit while the blue lines are the data points. The overall fitting parameter based on weighted agreement factoris $R_{wp} = 1.49\%$. The fitting is best in sample G but not so perfect in L. The agreement factors aregenerally low due to the high background. Similar analysis has been made for the other samples. All the instrumental and sample widths plots generally follow the same pattern as reported in literature, the FWHM falls with increasing 2ϴ. The Gaussian+Lorentziangenerally have the same features and shows that it is significant for sample L.
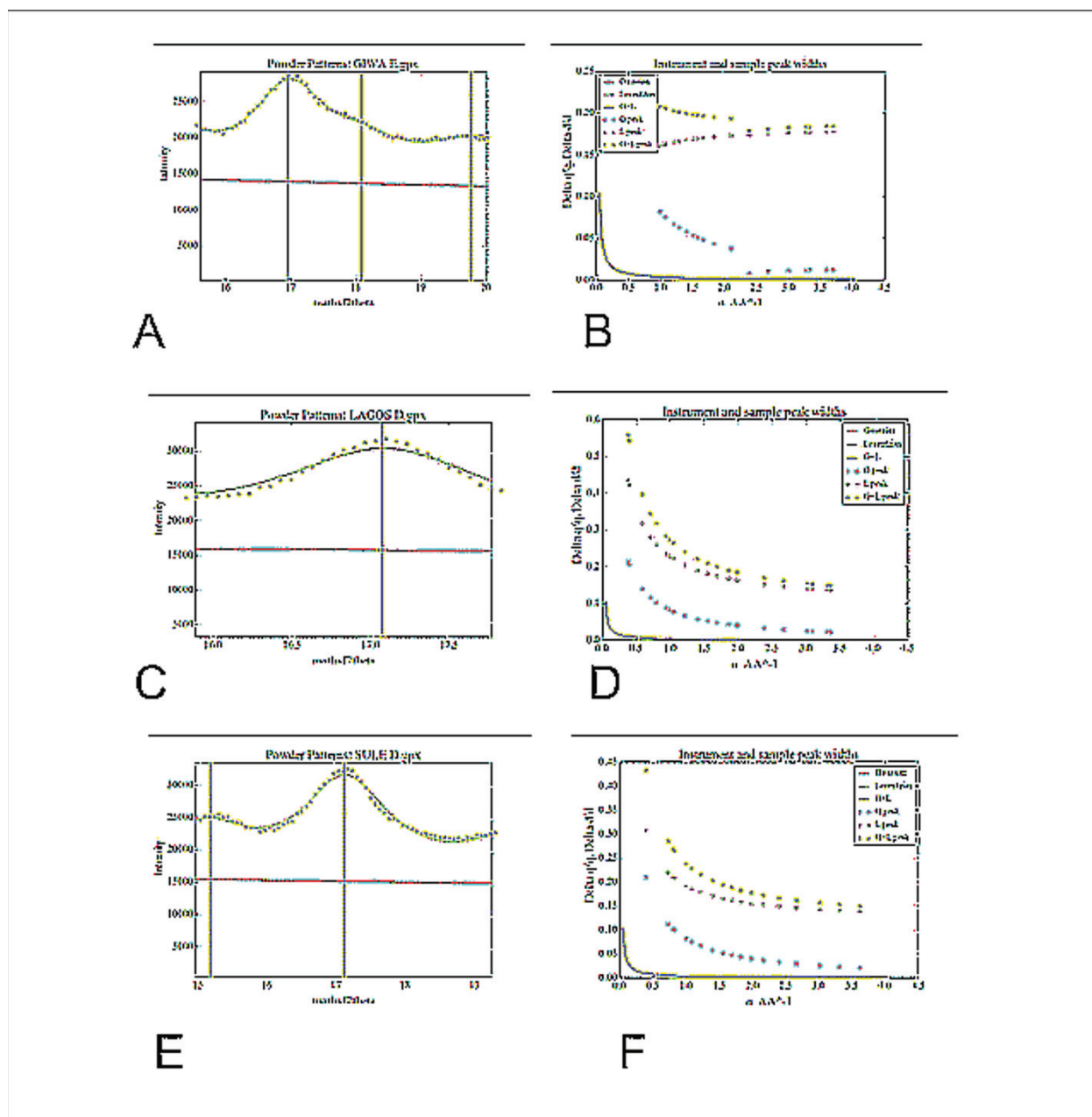
Figure 4. Profile fitting for peak of strongest intensities and Gaussian and Lorentzian fitsof instrumental profilesfor samples G, L and S. The fitting for L is shown in Figure 4(C,D) with $R_{wp} = 2.66\%$ together with the corresponding Gaussian and lorentzian broadenings. Figure 4(E, F) profile fitting for one peak and Gaussian and Lorentzian fits of instrumental profiles for sample L.The agreement factor is $R_{wp} = 1.75\%$.

Indexing was carried out on the XRD data by including peaks of small intensitiesin some cases to obtain at least M20 required for successful indexing. Table 5 shows the indexing results usingCMPR program (Brian, 2010) and the refined lattice parameters of the unit cells. All the cells were indexed on orthorhombic lattice with fairly high M(20) and F values for six strong reflections.

Table5. Indexing results based on orthorhombic symmetry and lattice parameters for three species of _D.R._

Species	h k l	F(6)	M(6)	a Å	b Å	c Å	$\alpha°$	$\beta°$	$\gamma°$
Lagos	120	80	20	8.0635	16.893	7.449	90	90	90
	111								
	031								
	200								
	002								
	132								
Giwa	200	200	50	11.742	15.689	6.245	90	90	90
	111								
	121								
	031								
	230								
	221								
Sule	110	224	56	7.624	11.158	5.803	90	90	90
	001								
	011								

120
021
030

6. Conclusion

Three starch samples were studied from the *dioscorearotundata* species:*Giwa*, *Sule and Lagos.* Proximate analysis shows that the moisture and amylose contents are relatively lower than reported. Similarly, carbohydrate found in this work is relatively high. The low moisture content implythe species have stability/longer storage time.

The particle sizes of the *D.R.*species are generally similar and oval in shape. Grains of relatively smaller and narrower distribution of sizes were obtained. The shapes and small sizesof the grains may have implications for applications in the paper, textile and cosmetic industry.

Elemental compositions of the *D.R.* species are generally the same with the most important finding being the presence of the element Ru, which is found in major concentrations (up to ~27%) and K (up to ~21%). The element Ru is appliedin the electronic industry as an optical component. The work suggests the presence of some important mineral deposits in the soil.

The starches exhibited the characteristic features of tuber starches and are all of the B-type, except *Giwa* and *Lagos* which are mixtures of B- and C-types. The crystallinity varied between 18-29% and while thecrystallite sizesare 2.5, 3.0 and 2.4nm, respectively, for *Giwa*, *Lagos* and *Sule*) and reflect the broadenings observed in the XRD patterns which are attributed to sample size. All the species of *D.R.* were indexedon orthorhombic lattice.With appropriate modifications of the native starch, *Lagos*

may find applications as pharmaceutical excipients and al, the species exhibit relatively competitive grain sizes that may compete with maize potato starches.

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