

# *In situ* dynamic rheological analysis of raw yam tubers: a potential phenotyping tool for quality evaluation

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## Abstract

**Background:** Most rheological analyses in yam have been done on starch gels, which requires starch extraction from the tubers. *In situ* rheology bypasses the need of starch extraction and relies on the original cell structure and complex matrix organization under stress or strain. Dynamic rheological properties of tuber from 16 accessions belonging to four yam species (*Dioscorea rotundata*, *D. alata*, *D. bulbifera* and *D. dumetorum*) were investigated for potential use as a medium throughput phenotyping screening tool that can indicate the quality of yam food products or their industrial potentials.

**Results:** Rheographs of the tubers illustrated differences in the structure of *D. bulbifera* compared to other yam species. High initial storage modulus ( $G'$ ) of yam parenchyma indicated tubers with strong and rigid structure which do not lose their structural integrity easily on heating. *Dioscorea rotundata* and *D. alata* varieties exhibited a lower temperature at which gelatinization took place ( $T_{gel}$ ) equivalent to the irreversible transition during starch gelatinization (75.3 and 79.8 °C) and took shorter time (867 and 958 s, respectively) to reach the  $G'$  maximum, compared to other species. The stress relaxation test showed that the higher the dry matter of the tubers, the higher the work to rupture the structure.

**Conclusion:** Rheological characteristics  $G'$ , loss modulus ( $G''$ ), swelling capacity and  $T_{gel}$  showed potential as suitable quality indicators for yam products. *In situ* rheological characterization of yam tubers could be used as an instrumental screening tool to phenotype for quality in yam products.

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**Keywords:** *Dioscorea*; viscoelastic properties; high-throughput screening; consumer preferences; efficient breeding

## INTRODUCTION

Yam tubers are edible starchy products of cultural, economic and nutritional importance, especially in the West African yam belt (Nigeria, Ghana, Côte D'Ivoire, Togo) and Ethiopia.<sup>1,2</sup> Yam is classified as the fourth most important root and tuber crop after potatoes, cassava and sweet potatoes.<sup>3</sup> Starch from other root and tuber crops, cassava, potatoes, sweet potatoes, is utilized extensively as an ingredient in both food and non-food industries. Though starch accounts for about 85% of the total dry weight of the yam tuber,<sup>4</sup> the industrial potential of yam starch is unexploited.<sup>5,6</sup> Exploration of the industrial potential requires suitable raw materials and rapid screening protocols to identify varieties with specific quality traits. Otegbayo *et al.*<sup>5</sup> studied the starch characteristics of some Nigerian yams and reported variability in their functional properties that could offer advantages for their industrial utilization. Several studies are reported<sup>4,7,8</sup> and ongoing research (RTBfoods project <https://rtbfoods.cirad>)<sup>9</sup> is being carried out on quality indicators in yam tubers that can predict the quality of preferred yam food products by consumers and medium to high-throughput methods that can be applied by breeders during

selection. For example, Ehouou *et al.*<sup>4</sup> reported the use of near-infrared spectroscopy (NIRS) to predict the chemical composition of yam tubers and textural quality of pounded yam.

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Rheology, the study of flow behavior of fluids and deformation behavior of solids<sup>10</sup> can provide information about mechanical, structural and viscoelastic properties of starch-concentrated systems. It can quantify both the viscous-like [loss modulus ( $G''$ )] and the elastic-like [storage modulus ( $G'$ )] properties of a material at different timescales. The  $G'$  is a measure of energy stored in the material during the shear process, it represents the elastic behavior of the sample. The  $G''$  value is a measure of the energy used, dissipated, or lost per cycle of sinusoidal deformation; it represents the viscous behavior of the material.<sup>11</sup> Loss tangent ( $\tan\delta$ ), or damping factor, is the quotient of the loss modulus to the storage modulus, which is the ratio of the viscous and the elastic portion of the viscoelastic deformation.

The rheological properties of raw Taewa potato were characterized and used as a tool to develop and evaluate chef ready Taewa products.<sup>12</sup> The uniaxial mechanical evolution of the  $G'$  and  $G''$  values has been evaluated for raw yam small pieces during short time cooking using a dynamic mechanical analyzer.<sup>13</sup> Viscosity, shear stress and shear rate have been used to measure functionalities of starch nanoparticles, and for product development, shelf-life estimation, sensory assessment and evaluation of the stability of the food products.<sup>14–16</sup> Ma *et al.*<sup>17</sup> applied rheology to study the potential of *Dioscorea opposita* starch as a thickener in the food industry. The potential of rheology to predict sensory textural quality and to screen samples for trained panelist evaluation has also been reported.<sup>18</sup> Thus, dynamic oscillatory properties of food products could be a useful tool to identify quality indicators and applied in product development.

Most reported studies involving rheological analyses in yam<sup>19–21</sup> were usually done on the starch gel, which entailed starch isolation. This time-consuming stage involving the quantitative extraction of starch remains a challenge. Characterizing the *in situ* rheological properties of raw yam tubers might help to address this issue because the original cell structure (raw yam) will be used. Thus, in this study we investigated whether rheological screening protocol can be applied on samples of raw tuber parenchyma to predict the quality of yam food products. We focused on characterizing the dynamic rheological properties of raw yam tubers from 16 varieties of four commonly cultivated species [*Dioscorea rotundata* (white yam), *Dioscorea alata* (water yam), *Dioscorea bulbifera* (aerial or potato yam) and *Dioscorea dumetorum* (bitter yam)] in order to determine and monitor changes in their mechanical, structural, textural and viscoelastic properties during heating, and the potential of using their rheological profile or characteristics as a medium throughput phenotyping (MTTP) tool that can help to quantify intrinsic attributes which can be employed to predict the quality of yam food products and select breeding lines for consumer preferences.

## MATERIALS AND METHODS

### Materials

Sixteen varieties of yam (five varieties each of *D. rotundata*, *D. alata* and *D. bulbifera* and one variety of *D. dumetorum*), from the International Institute of Tropical Agriculture (IITA), Ibadan (Nigeria) yam germplasm collection were used for this study (Fig. 1).

### Methods

#### Starch extraction, dry matter content and yam flour processing

Dry matter of fresh tubers was determined in an air-drying oven at 105 °C until constant weight was attained.<sup>22</sup> Starch was extracted

from the yam tubers as described in Bolanle *et al.*<sup>23</sup> The analyses were done in triplicate. For the yam flour, the tubers were peeled, washed, diced into cubes, and dried in an air convection oven at 40 °C for 72 h. The dried chips were milled into flour (to pass through a mesh of 250  $\mu\text{m}$ ) and packed into zip-lock bags.

### Analyses on yam tubers

#### Dynamic rheology

The dynamic rheological properties of the raw tubers were determined by modification of the published protocol.<sup>12</sup> About 1 mm thick slice of yam parenchyma was cut transversely from the middle of the tuber by means of ham cutter; then a cork borer (inner diameter 20.9 mm) was used to cut out cylindrical disks from the slice. The samples were then hermetically stored in plastic containers at room temperature. Rheological analysis was performed with a rheometer (Physica MCR 301; Anton Paar GmbH, Graz, Austria) coupled with a serrated flat probe (PP25, 25 mm probe). Prior to temperature sweep at fixed normal force 3.5 N, 7% strain and 1 Hz frequency with automatic adjustment of the gap, the linear viscoelastic region for each genotype was determined by normal force sweep (0.5–100 N), strain sweep (0.01–100%) and frequency sweep (0.1–100 Hz). A thin smear of silicone oil was used to cover the edge of the samples to prevent evaporation. The samples were then heated from 35 to 90 °C at 0.05 °C/s and held for 720 s at 90 °C and cooled from 90 to 25 °C (at about 0.35 °C/s). Computation of results was done with RheoPlus/32 software version 2.65 (Anton Paar GmbH). The dynamic oscillation parameters evaluated were:  $G'$ ,  $G''$ , and  $\tan\delta$ . The analyses were done in triplicate. The  $G'_{\text{max}}$  represents the maximum  $G'$  during the heating cycle while the  $G'_{\text{min}}$  represents the minimum  $G'$ . SC represents the swelling capacity of the starches, TS is time taken for the starch granule to swell, TP is time taken by the starch gel to rupture, and  $T_{\text{gel}}$  is the temperature at which gelatinization took place. The values of  $G'_{\text{max}}$ , SC, TS, TP and  $T_{\text{gel}}$  were derived from the rheological profile of the tubers (Fig. 2(a)).

#### Relaxation properties of the tuber

Complementary analysis on the relaxation properties of the raw tubers to determine the relaxation work (work done in breaking the structure of the tuber) and the ratio of the initial to final relaxation force was performed on the raw tubers by means of TA-Xt Plus texture analyzer (Stable Micro Systems, Godalming, UK). Automatic computation was conducted using the Texture Exponent software. Cylindrical disks were cut out from the central core of the fresh tubers using a core borer (diameter 20.9 mm and 15 mm thick). The samples were placed on the platform of the texture analyzer and subjected to a 20% uniaxial strain (trigger force  $5 \times g$ ) at test speed of 1 mm/s by means of a cylindrical probe (58.85 mm diameter). The uniaxial compression was held for 60 s for tuber relaxation. The probe was removed at 10 mm/s. The maximum force (in newtons) during the holding stage and its corresponding work (in newton-second) were computed. The test was replicated eight times for each sample.

### Analyses of yam flour

#### Starch content

The starch content of the flour samples was determined by the enzymatic procedure.<sup>24</sup> This method is based on enzymatic hydrolysis of starch to glucose by a system of oxido-reduction enzymes; amyloglucosidase, glucose oxidase and peroxidase. The starch concentration was calculated as follow:



**TDb 3048**

**TDb 3059**

**TDb 3084**

**TDb 3884**

**TDb 3069**

(a) *D. bulbifera* varieties



**Amula**

**TDr 99-15**

**TDr 89/02665**

**Baidza**

**TDr 93-31**

(b) *D. rotundata* Varieties



**TDa 01/00012**

**TDa 93-36**

**TDa 92-2**

**TDa 297**

**TDa 291**

(c) *D. alata* Varieties



(d) *D. dumetorum*

**Figure 1.** Photographs of tubers from the different yam species characterized.

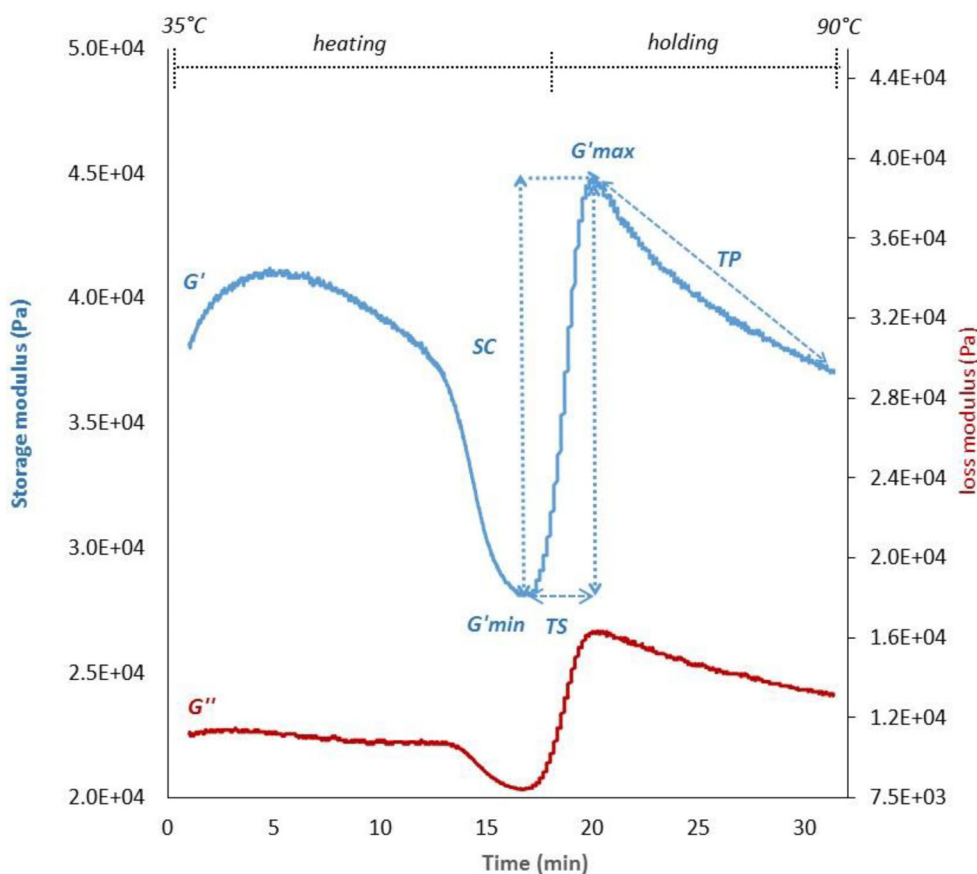
$$\text{Percentage starch} = \frac{\text{absorbance} \times \text{slope} \times \text{dilution factor} \times 0.9}{\text{sample weight} \times \text{dry matter}}$$

**Analyses of yam starch**

*Viscoamylograph characteristics of yam starch*

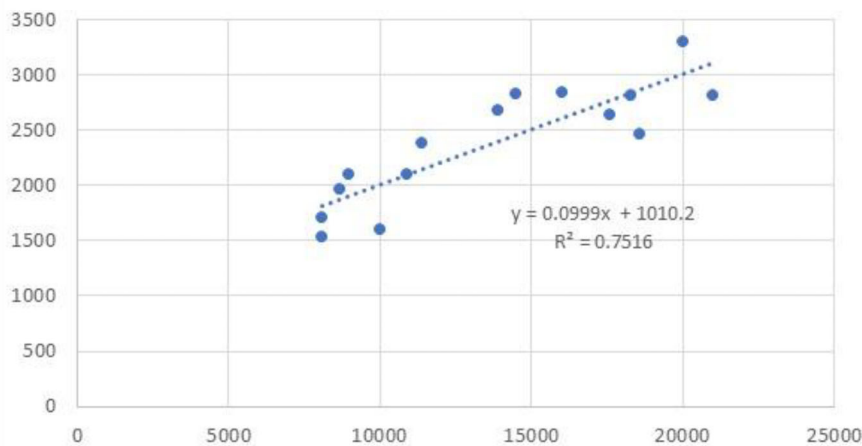
Hot starch dispersion viscosity profiles were determined using a Physica MCR 301 rheometer (Anton Paar, Ostfildern

Germany) fitted with a starch cell and starch stirrer (ST24-2D/2 V/2 V-30). Starch slurry at 8% concentration (w/v dry basis) was first stirred at 960 rpm for 10 s at 50 °C prior to a 160 rpm stirring for 60 s at 50 °C as reported by Pérez et al.<sup>25</sup> The following viscoamylograph properties were determined: pasting temperature (PT), peak viscosity (PV), hot paste viscosity (HPV), cool paste viscosity (CPV), and breakdown viscosity (BD).



(a)

HPV vs SP



(b)

**Figure 2.** (a) Typical rheological temperature sweep profile of a yam tuber.  $G'_{max}$ : maximum storage modulus ( $G'$ ),  $G'_{min}$ : minimum  $G'$ , TS: time taken for gel to swell, TP is time take for gel to rupture, SP: swelling power (b) Correlation between hot paste viscosity (HPV, viscosity related to maximum granule swelling at plateau) and swelling capacity (SC).

**Statistical analyses**

The data generated were analyzed by the SAS package (Statistical Analysis Systems of SAS Institute, Cary, NC, USA) Analysis of variance and means separations were calculated by the general linear model (GLM) procedure.

**RESULTS**

**Specific gravity, dry matter, and starch content of the tubers**

Dry matter and starch content of the tubers are presented in Table 1. There were both intra-species (differences between

**Table 1.** Dry matter and starch content of *Dioscorea* tubers<sup>†</sup>

Species cultivar	Varieties <sup>‡</sup>	Dry matter (g/kg) <sup>§</sup>	Starch content (g/kg)
<i>Dioscorea alata</i>	TDa 01/00012	29.98 <sup>c</sup>	13.01 <sup>b</sup>
	TDa 92-2	26.23 <sup>b</sup>	12.81 <sup>b</sup>
	TDa 93-36	24.38 <sup>a</sup>	15.28 <sup>c</sup>
	TDa 291	26.92 <sup>b</sup>	10.44 <sup>a</sup>
	TDa 297	30.50 <sup>c</sup>	15.07 <sup>c</sup>
	Mean ± standard deviation <sup>¶</sup>	27.60 ± 2.24 <sup>a</sup>	13.33 ± 1.97 <sup>a</sup>
<i>Dioscorea bulbifera</i>	TDb 3048	30.76 <sup>c</sup>	15.17 <sup>c</sup>
	TDb 3059	31.04 <sup>c</sup>	14.80 <sup>c</sup>
	TDb 3069	28.08 <sup>b</sup>	11.83 <sup>b</sup>
	TDb 3084	21.01 <sup>a</sup>	12.21 <sup>b</sup>
	TDb 3884	22.77 <sup>a</sup>	9.48 <sup>a</sup>
	Mean ± standard deviation <sup>¶</sup>	26.73 ± 4.61 <sup>a</sup>	12.69 ± 2.34 <sup>a</sup>
<i>Dioscorea dumetorum</i>	<i>Esuru funfun</i>	28.18 <sup>b</sup>	15.77 <sup>b</sup>
<i>Dioscorea rotundata</i>	TDr Amula	36.01 <sup>c</sup>	20.45 <sup>c</sup>
	TDr Baidza	31.95 <sup>a</sup>	15.52 <sup>a</sup>
	TDr 89/2665	34.61 <sup>b</sup>	20.62 <sup>c</sup>
	TDr 93-31	37.65 <sup>d</sup>	22.38 <sup>d</sup>
	TDr 99-15	32.90 <sup>a</sup>	17.78 <sup>b</sup>
	Mean ± standard deviation <sup>¶</sup>	34.62 ± 2.30 <sup>c</sup>	19.35 ± 2.70 <sup>c</sup>

<sup>†</sup> Means with the same superscript lowercase letters in the same column under each species are not significantly different ( $P \leq 0.05$ ).

<sup>‡</sup> TDa, tropical *Dioscorea alata*; TDb, tropical *Dioscorea bulbifera*; TDr, tropical *Dioscorea rotundata*.

<sup>§</sup> Data on fresh weight basis.

<sup>¶</sup> Significant differences between the means among the yam species.

**Table 2.** Viscoamylograph properties of yam starch<sup>†</sup>

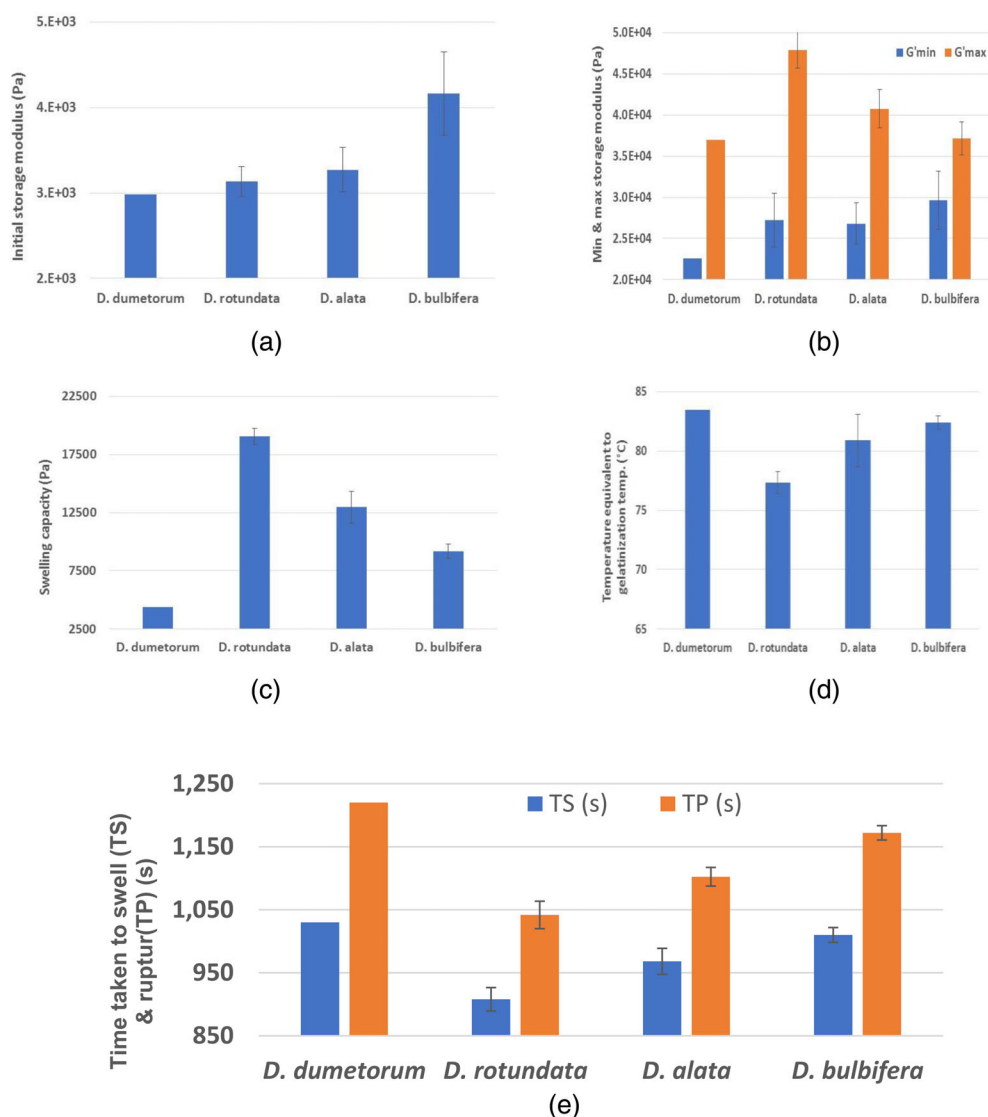
Species	Variety <sup>‡</sup>	PT (°C)	PV (cP)	HPV (cP)	CPV (cP)	BD (cP)
<i>Dioscorea alata</i>	TDa 01/00012	84.5 <sup>c</sup>	3961 <sup>b</sup>	2842 <sup>a</sup>	3732 <sup>c</sup>	1120 <sup>c</sup>
	TDa 92-2	85.9 <sup>a</sup>	3094 <sup>e</sup>	2825 <sup>b</sup>	3745 <sup>b</sup>	269 <sup>e</sup>
	TDa 291	84.9 <sup>b</sup>	3103 <sup>d</sup>	2683 <sup>c</sup>	3873 <sup>a</sup>	420 <sup>d</sup>
	TDa 297	82.6 <sup>e</sup>	3733 <sup>c</sup>	2093 <sup>e</sup>	2853 <sup>e</sup>	1640 <sup>b</sup>
	TDa 93-36	83.9 <sup>d</sup>	4525 <sup>a</sup>	2377 <sup>d</sup>	3278 <sup>d</sup>	2149 <sup>a</sup>
	Mean ± standard deviation <sup>§</sup>	84.36 ± 1.21 <sup>a</sup>	3683 ± 573 <sup>a</sup>	2564 ± 305 <sup>a</sup>	3496 ± 403 <sup>ab</sup>	1119 ± 752 <sup>a</sup>
<i>Dioscorea bulbifera</i>	TDb3048	85.7 <sup>e</sup>	1798 <sup>c</sup>	1703 <sup>c</sup>	3133 <sup>c</sup>	95 <sup>a</sup>
	TDb3059	86.6 <sup>c</sup>	2111 <sup>a</sup>	2099 <sup>a</sup>	3984 <sup>a</sup>	12 <sup>d</sup>
	TDb3069	86.4 <sup>d</sup>	2019 <sup>b</sup>	1959 <sup>b</sup>	3314 <sup>b</sup>	60 <sup>b</sup>
	TDb3084	86.7 <sup>b</sup>	1624 <sup>d</sup>	1529 <sup>e</sup>	2709 <sup>d</sup>	95 <sup>a</sup>
	TDb3884	87.2 <sup>a</sup>	1624 <sup>d</sup>	1605 <sup>d</sup>	2536 <sup>e</sup>	19 <sup>c</sup>
	Mean ± standard deviation <sup>§</sup>	86.5 ± 0.5 <sup>b</sup>	1835 ± 213 <sup>b</sup>	1779 ± 229 <sup>b</sup>	3135 ± 537 <sup>a</sup>	56 ± 39 <sup>b</sup>
<i>Dioscorea dumetorum</i>	<i>Esuru funfun</i>	85.0 ± 0.2 <sup>ab</sup>	1091 ± 4 <sup>b</sup>	443 ± 6 <sup>c</sup>	604 ± 6 <sup>c</sup>	649 ± 11 <sup>ab</sup>
<i>Dioscorea rotundata</i>	TDr89/02665	82.6 <sup>e</sup>	3548 <sup>c</sup>	2808 <sup>c</sup>	4228 <sup>b</sup>	740 <sup>b</sup>
	TDr93-31	83.8 <sup>b</sup>	3294 <sup>d</sup>	2644 <sup>d</sup>	3839 <sup>d</sup>	650 <sup>d</sup>
	TDr9915	84.7 <sup>a</sup>	3133 <sup>e</sup>	2462 <sup>e</sup>	3448 <sup>e</sup>	672 <sup>c</sup>
	TDr Amula	82.7 <sup>d</sup>	3628 <sup>b</sup>	3302 <sup>a</sup>	4618 <sup>a</sup>	323 <sup>e</sup>
	TDr Baidza	83.1 <sup>c</sup>	4435 <sup>a</sup>	2818 <sup>b</sup>	4095 <sup>c</sup>	1618 <sup>a</sup>
	Mean ± standard deviation <sup>§</sup>	83.3 ± 0.8 <sup>a</sup>	3608 ± 477 <sup>a</sup>	2752 ± 253 <sup>a</sup>	3982 ± 382 <sup>b</sup>	800 ± 460 <sup>ab</sup>

Abbreviation: PT, pasting temperature; PV, peak viscosity; HPV, hot paste viscosity; CPV, cool paste viscosity; BD, breakdown viscosity.

<sup>†</sup> Means with the same superscripts in the same column under each species are not significantly different ( $P \leq 0.05$ ).

<sup>‡</sup> TDa, tropical *Dioscorea alata*; TDb, tropical *Dioscorea bulbifera*; TDr, tropical *Dioscorea rotundata*.

<sup>§</sup> Significant differences between the means among the yam species.



**Figure 3.** (a) Initial storage modulus; (b) minimum and maximum storage moduli; (c) swelling capacity; (d) initial storage modulus; (d) summary of temperature equivalent to the gelatinization; (e) time taken to swell (TS) and time to gel rupture (TP) during *in situ* oscillatory rheological profiling of raw yam tubers.

varieties in a species) and inter-species (differences between the species) differences in terms of the dry matter content. The mean dry matter of the tubers among the species ranged from 26 to 34.6 g kg<sup>-1</sup> with *D. rotundata* having the highest mean dry matter (34.6%) compared with the other species. There was no significant difference ( $P < 0.05$ ) between the mean dry matter of *D. alata* and *D. bulbifera*. The order of the starch content among the *Dioscorea* species was: *D. rotundata* > *D. dumetorum* > *D. alata* > *D. bulbifera*, though there were significant differences among the varieties in terms of their starch contents. A positive correlation was observed between the dry matter and the starch content ( $r = 0.91$ ) of the four yam species.

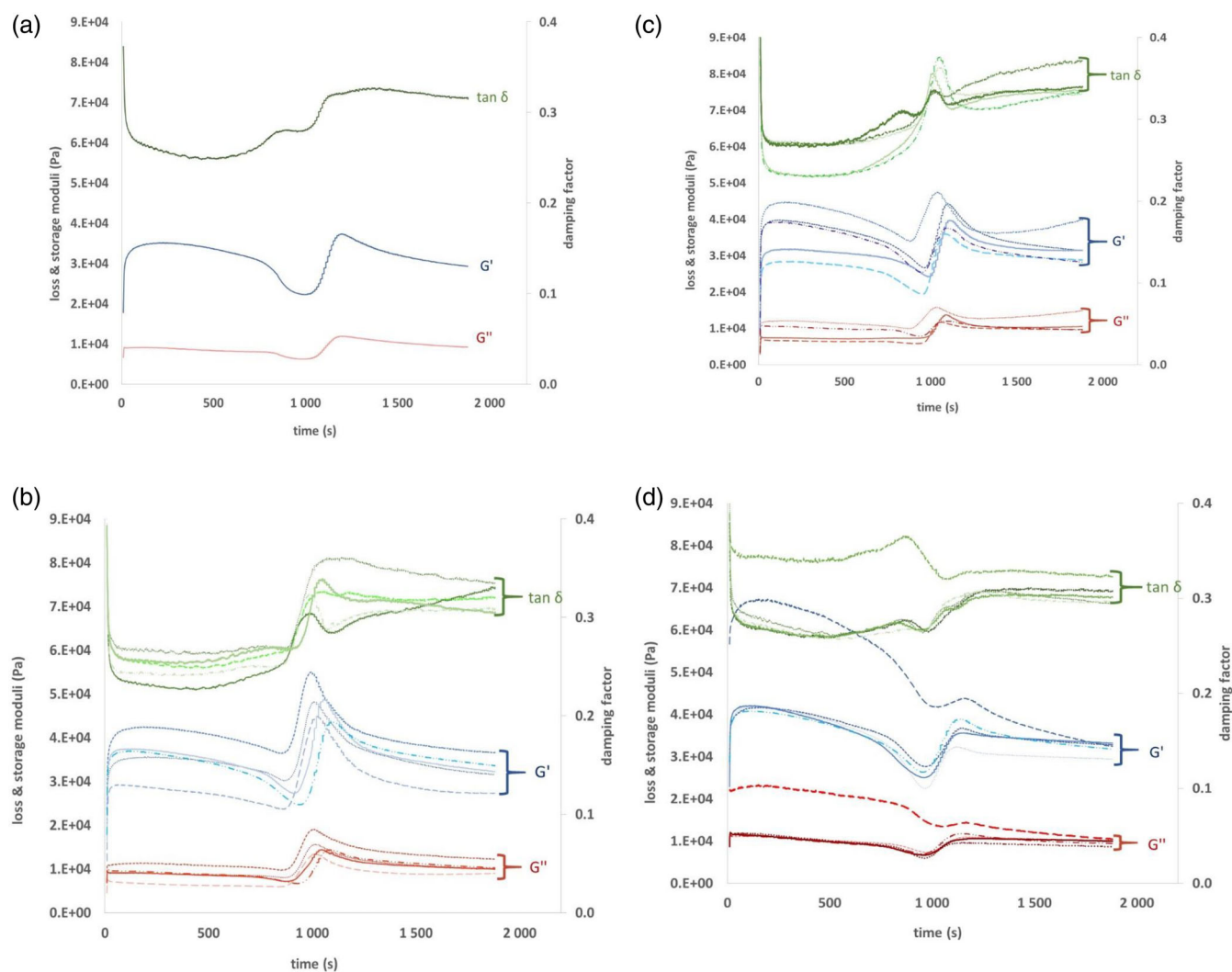
### Viscoamylograph characteristics of yam starch

The viscoamylograph properties of the yam starch are presented in Table 2. There were significant differences ( $P < 0.05$ ) among species and among varieties within each species in terms of their viscoamylograph characteristics. The PT of the yam varieties ranged between 82.6 and 87.2°C with *D. bulbifera* species having the highest among the four species. There was no significant

difference in the PV, HPV, CPV and the PT of *D. alata* and *D. rotundata* species. *Dioscorea dumetorum* and *D. bulbifera* had the least PV and HPV among the yam species. *Dioscorea rotundata* had the highest CPV amongst the yam species while *D. dumetorum* had the lowest. *Dioscorea dumetorum* had low maximum PV and low HPV at the plateau. *Dioscorea bulbifera* varieties exhibited resistance to shear at the plateau, and a significant increase in the final viscosity on cooling. There were significant differences in the pasting characteristics of the yam varieties within each species.

### Rheological properties of raw tubers

The typical rheological profile of the tubers during heating, holding and cooling stages is illustrated in Fig. 2(a), the graphical representation of parameters extracted from the rheological profile [ $G'$  (initial, minimum and maximum), hypothesized SC, TS, TP and  $T_{gel}$  is the temperature at  $G'_{max}$ ] is presented in Fig. 3(a)–(e) while Fig. 4(a)–(d) shows the rheological profile of the different yam species. At the initial heating of the tubers, there was a



**Figure 4.** (a) Rheological profile of yam parenchyma of *Dioscorea dumetorum* tuber. (b) Rheological profile of yam parenchyma from five varieties of *D. rotundata* tubers. (c) Rheological profile of yam parenchyma from five varieties of *D. alata* tubers. (d) Rheological profile of yam parenchyma of five varieties of *D. bulbifera* tubers.

general decrease in the  $G'$  up to a certain time; then it increased with further heating to a maximum ( $G'_{\max}$ ) and then decreased on cooling. The  $G'_{\max}$  is the maximum  $G'$  during the heating cycle while the  $G'_{\min}$  represents the minimum  $G'$  (destruction of the starch gel as a consequence of prolonged heating). *Dioscorea rotundata* exhibited the highest storage moduli ( $G'_{\max}$ ) among the three species with an average of 47 900 Pa followed by *D. alata* (40 760 Pa), while *D. bulbifera* and *D. dumetorum* have a similar maximum  $G'$  (37 140 and 37 000 Pa), respectively (Table 3). The temperature at which the  $G'_{\max}$  reached  $T_{\text{gel}}$ , varied among the yam species and within the varieties of a species. *Dioscorea rotundata* varieties had a lower  $T_{\text{gel}}$  (75.3–79.8°C) and took a shorter time (TS) (867–958 s) to reach the  $G'_{\max}$  compared to other species. Among the *D. rotundata* cultivars, TDr 89/02665 had the highest  $G'_{\max}$  and the lowest  $T_{\text{gel}}$  at the shortest time (867 s). This trend was also observed among the *D. alata* cultivars with TDa 297 having the highest  $G'_{\max}$  at the lowest  $T_{\text{gel}}$  this trend was not observed among *D. bulbifera* cultivars. All the *D. bulbifera* cultivars started with remarkably high initial  $G'$  before decreasing to a minimum and then increasing to the  $G'_{\max}$  (Fig. 4).

Among the *D. bulbifera* species, TDb 3884 exhibited the highest initial  $G'$  (6300 Pa). The  $T_{\text{gel}}$  of this species was the highest (81.6–84.3 °C). The amplitude of storage modulus between the  $G'_{\min}$  and  $G'_{\max}$  [the difference in height between the minimum  $G'$  (the  $G'$  after the loss of structural integrity) and the  $G'_{\max}$ ] was hypothesized to be an indicator of SC of the starch in the tubers. This is because this inter-phase represents the swelling of the starch granules to fill the whole volume. SC of the starches in the tuber were, in order: *D. rotundata* > *D. alata* > *D. bulbifera* > *D. dumetorum*. *Dioscorea rotundata* had an average of 19 100 Pa, with the highest from var. Baidza, while TDb 3884 had the lowest (1000 Pa) among all the yam cultivars (Table 3, Fig. 3(a)–(e)). There was a correlation ( $r = 0.93$ ) between  $T_{\text{gel}}$  (hypothesized gelatinization temperature of the starch in the raw yam tuber) and PT and HPV of the yam starch (viscosity related to maximum granule swelling at plateau) and SC (extracted from the rheological profile of the raw yam tuber) (Fig. 2(b)). From the rheographs (Fig. 4(a)–(d)),  $G'$  values remained higher than those of  $G''$  in all cultivars. The time taken for the starch granule to swell is represented by TS, while TP depicts the time taken by the starch gel to rupture. These parameters (for each species)

**Table 3.** Storage moduli, swelling power, gelatinization temperature and time taken during oscillatory rheological profiling of raw yam tuber<sup>†</sup>

Species	Varieties <sup>‡</sup>	Initial $G'$ (Pa)	Minimum $G'$ ( $G'_{\min}$ ) (Pa)	Maximum $G'$ ( $G'_{\max}$ ) (Pa)	Time taken to swell (TS) (s)	Time taken for gel rupture (TP) (s)	Temperature at which gelatinization took place ( $T_{\text{gel}}$ ) (°C)	Swelling Capacity (SC) (Pa)
<i>Dioscorea dumentorum</i>	<i>Esuru funfun</i>	2980 <sup>a</sup>	22600 <sup>a</sup>	37000 <sup>a</sup>	1030 <sup>a</sup>	1220 <sup>a</sup>	83.5 <sup>c</sup>	4400 <sup>a</sup>
	TDr 89/02665	3540 <sup>c</sup>	36700 <sup>c</sup>	55000 <sup>d</sup>	867 <sup>a</sup>	989 <sup>a</sup>	75.3 <sup>e</sup>	18300 <sup>c</sup>
	TDr 93-31	2996 <sup>b</sup>	30500 <sup>bc</sup>	48100 <sup>bc</sup>	886 <sup>a</sup>	1020 <sup>ab</sup>	76.2 <sup>d</sup>	17600 <sup>c</sup>
	TDr 99-15	3280 <sup>bc</sup>	25000 <sup>ab</sup>	43600 <sup>a</sup>	958 <sup>b</sup>	1100 <sup>d</sup>	79.8 <sup>a</sup>	18600 <sup>bc</sup>
	TDr Baidza	2620 <sup>a</sup>	23800 <sup>a</sup>	44800 <sup>abc</sup>	895 <sup>a</sup>	1030 <sup>bc</sup>	78.6 <sup>b</sup>	21000 <sup>a</sup>
	TDr Amula	3230 <sup>bc</sup>	20000 <sup>a</sup>	48000 <sup>c</sup>	934 <sup>b</sup>	1070 <sup>cd</sup>	76.7 <sup>c</sup>	20000 <sup>ab</sup>
	Mean <sup>§</sup>	3133.2 <sup>ab</sup>	27200 <sup>b</sup>	47900 <sup>c</sup>	908 <sup>a</sup>	1041.8 <sup>a</sup>	77.2 <sup>a</sup>	19100 <sup>c</sup>
	SE	154.69	2910.15	1981.92	16.60	19.47	0.82	614.82
	TDa 291	2500 <sup>a</sup>	21000 <sup>a</sup>	36000 <sup>a</sup>	980 <sup>bc</sup>	1090 <sup>b</sup>	80.9 <sup>b</sup>	13900 <sup>c</sup>
	TDa 001/0012	3490 <sup>c</sup>	28000 <sup>b</sup>	44000 <sup>bc</sup>	987 <sup>bc</sup>	1111 <sup>b</sup>	81.0 <sup>b</sup>	16000 <sup>a</sup>
<i>Dioscorea alata</i>	TDa 297	3780 <sup>c</sup>	34500 <sup>c</sup>	47000 <sup>c</sup>	901 <sup>a</sup>	1060 <sup>a</sup>	75.0 <sup>d</sup>	9000 <sup>e</sup>
	TDa 92-2	2980 <sup>b</sup>	25000 <sup>ab</sup>	39800 <sup>ab</sup>	1010 <sup>c</sup>	1111 <sup>b</sup>	87.5 <sup>a</sup>	14500 <sup>b</sup>
	TDa 93-36	3610 <sup>c</sup>	25600 <sup>b</sup>	37000 <sup>a</sup>	963 <sup>b</sup>	1140 <sup>c</sup>	80.1 <sup>c</sup>	11400 <sup>d</sup>
	Mean <sup>§</sup>	3272 <sup>b</sup>	26820 <sup>b</sup>	40760 <sup>b</sup>	968.2 <sup>a</sup>	1102.4 <sup>a</sup>	80.9 <sup>b</sup>	12960 <sup>b</sup>
	Standard error	234.64	2225.40	2087.49	18.42	13.25	4.2	1237.17
	TDb 3069	3680 <sup>a</sup>	28000 <sup>a</sup>	36700 <sup>b</sup>	994 <sup>a</sup>	1160 <sup>a</sup>	81.6 <sup>b</sup>	8700 <sup>c</sup>
	TDb 3084	3890 <sup>a</sup>	23900 <sup>a</sup>	32000 <sup>a</sup>	1000 <sup>a</sup>	1150 <sup>a</sup>	82.0 <sup>b</sup>	8100 <sup>c</sup>
	TDb 3884	5890 <sup>b</sup>	42000 <sup>b</sup>	43000 <sup>c</sup>	10500 <sup>b</sup>	1210 <sup>b</sup>	84.3 <sup>a</sup>	1000 <sup>b</sup>
	TDb 3048	3670 <sup>a</sup>	26900 <sup>a</sup>	35500 <sup>ab</sup>	10100 <sup>b</sup>	1170 <sup>b</sup>	82.5 <sup>b</sup>	8100 <sup>c</sup>
	TDb 3059	3680 <sup>a</sup>	27600 <sup>a</sup>	38500 <sup>b</sup>	994 <sup>a</sup>	1170 <sup>b</sup>	81.6 <sup>b</sup>	10900 <sup>a</sup>
Mean <sup>§</sup>	4162 <sup>c</sup>	29680 <sup>c</sup>	37140 <sup>a</sup>	4717.6 <sup>b</sup>	756.2 <sup>a</sup>	82.4 <sup>c</sup>	7360 <sup>a</sup>	
Standard error	433.97	3162.82	1809.59	2279.88	261.67	1.1	1671.41	

<sup>†</sup> Means with the same superscripts in the same column under each species are not significantly different ( $P \leq 0.05$ ).<sup>‡</sup> TDa, tropical *Dioscorea alata*; TDr, tropical *Dioscorea rotundata*; TDb, tropical *Dioscorea bulbifera*.<sup>§</sup> Significant differences between the means among the yam species.



**Table 4.** Relaxation properties of fresh yam tubers<sup>†</sup>

Species	Varieties <sup>‡</sup>	Work done (g.mm)	Force (g)
<i>Dioscorea alata</i>	TDa 01/00012	17835 <sup>a</sup>	15108 <sup>a</sup>
	TDa 92-2	20195 <sup>a</sup>	16639 <sup>a</sup>
	TDa 93-36	18368 <sup>a</sup>	15188 <sup>a</sup>
	TDa 291	18522 <sup>a</sup>	16227 <sup>a</sup>
	TDa 297	17057 <sup>a</sup>	16033 <sup>a</sup>
	Mean ± standard deviation <sup>§</sup>	18 395 ± 1158 <sup>a</sup>	15 839 ± 668 <sup>a</sup>
<i>Dioscorea bulbifera</i>	TDb 3048	13987 <sup>a</sup>	12066 <sup>a</sup>
	TDb 3059	11537 <sup>a</sup>	10765 <sup>a</sup>
	TDb 3069	13012 <sup>a</sup>	12334 <sup>a</sup>
	TDb 3084	11144 <sup>a</sup>	10344 <sup>a</sup>
	TDb 3884	10902 <sup>a</sup>	10595 <sup>a</sup>
	Mean ± standard deviation <sup>§</sup>	12 116 ± 1328 <sup>bc</sup>	11 220 ± 911 <sup>bc</sup>
<i>Dioscorea dumetorum</i>	<i>Esuru funfun</i>		
	Mean ± standard deviation <sup>§</sup>	11 182 ± 6548 <sup>b</sup>	10 255 ± 4209 <sup>b</sup>
<i>Dioscorea rotundata</i>	TDr Amula	15085 <sup>a</sup>	18594 <sup>c</sup>
	TDr Baidza	12100 <sup>a</sup>	10517 <sup>a</sup>
	TDr 89/2665	16457 <sup>ab</sup>	14791 <sup>b</sup>
	TDr 93-31	21144 <sup>bc</sup>	17335 <sup>bc</sup>
	TDr 99-15	23273 <sup>c</sup>	18594 <sup>c</sup>
	Mean ± standard deviation <sup>§</sup>	176 118 ± 4545 <sup>ac</sup>	15 966 ± 3419 <sup>ac</sup>

<sup>†</sup> Means with the same superscripts in the same column under each species are not significantly different ( $P \leq 0.05$ ).

<sup>‡</sup> TDa, tropical *Dioscorea alata*; TDb, tropical *Dioscorea bulbifera*; TDr, tropical *Dioscorea rotundata*.

<sup>§</sup> Significant differences between the means among the yam species.

are presented in Table 3 and Fig. 3(a)–(e). In all the yam varieties,  $\tan\delta$  decreased during cooling (90–25 °C) and it is smaller than unity (< 1) in all the yam varieties.

### Relaxation properties of the tuber

Stress relaxation was applied to characterize the work and the force applied to rupture the fresh yam tubers under constant strain and to differentiate the relaxation behavior among species and varieties (Table 4). Significant differences ( $P < 0.05$ ) were observed between computed work done and force to rupture the structure of the yam tubers between *D. rotundata*, *D. dumetorum* and *D. bulbifera* species respectively, whereas no significant difference existed between *D. rotundata* and *D. alata*.

## DISCUSSION

Positive correlation observed between the dry matter and the starch contents ( $r = 0.91$ ) of the four yam species implied that the higher the starch content the higher was the dry matter of the yam tubers. A similar trend was observed by Ehounou *et al.*<sup>4</sup> According to Zhu,<sup>2</sup> this is expected, since it has been reported that starch accounts for about 85% of the dry matter of the yam tubers.

Significant differences ( $P < 0.05$ ) observed among the species and varieties in terms of their viscoamylograph properties illustrates the variability and diversity in the thermal and functional properties of the tubers. PT is the temperature at the onset of rise in viscosity. *Dioscorea bulbifera* starch had the highest PT amongst the yam species with var TDb 3884 having the highest PT with 87.2°C. It was also observed that in the rheological profile of the

yam tubers, this variety (TDb 3884) had the highest  $T_{gel}$  (which was hypothesized as close to the gelatinization temperature). Thus, this suggests that  $T_{gel}$  in the raw tuber can be an indicator of the PT or of the gelatinization temperature of the starch and also imply that its food products will have a higher cooking time compared to those from other yam species. PV is the point at which gelatinized starch reaches its maximum viscosity during heating in water. The high PV and HPV of *D. rotundata* and *D. alata* varieties are an indication of their water binding capacities, their viscous load (as a result of viscosity of the starch) since they have an equilibrium between granule swelling which causes increase in viscosity and polymer leaching and also that their starches have resistance to swelling and granule rupture.<sup>26</sup> All the yam varieties showed an increase in viscosity on cooling, this is the CPV. Increase in the final viscosity (CPV) in all the yam varieties indicates the capacity of their starches to form a viscous paste or gel after cooking and cooling. This increase in final viscosity during cooling is usually as a result of re-association of swollen granules, or colloidal and molecularly dispersed starch molecules as the temperature decreased. It is also an indication of resistance of the starch to mechanical shear at high temperature. Thus, *D. rotundata* varieties which had the highest CPV amongst the yam varieties exhibited a restricted increase in the gel viscosity on cooling and may form a stronger gel compared to other species. Starches from *D. alata* species had the highest average BD among the yam species. Higher BD indicates a higher paste stability and resistant to mechanical fragmentation during shear. The implication of this to the yam starches is that the paste stability of the starches is the order *D. alata* > *D. rotundata* > *D. dumetorum* > *D. bulbifera*.

The rheological profiles exhibited by the raw tubers were similar to those of raw potatoes reported by Singh *et al.*<sup>12</sup> The decrease in the  $G'$  and  $G''$  at the beginning of the heating period (Fig. 2(a)) can be attributed to softening of the tuber due to loss of turgor and cellular disorganization which leads to loss of structural integrity or its rigidity/strength or loss in network aggregates.<sup>9,27,28</sup> As the heating progresses at a constant rate there was gradual increase in the  $G'$  as a result of swelling of the starch granules, crystalline melting, solubilization, and breaking of intramolecular hydrogen bonds. There was a corresponding increase in starch molecule–water hydrogen bonding with water, and a transition from the liquid system, with dispersed particles, to a system nearly packed with deformed particles.<sup>9,29,30</sup> After reaching a maximum ( $G_{\max}$ ), the  $G'$  gradually decreased due to destruction of the starch gel as a consequence of prolonged heating, melting of the remaining crystallites and partial separation and leaching of amylose and amylopectin. This decrease in  $G'$  caused the swollen starch granules to become soft, thus decreasing the elastic moduli. According to Keetels *et al.*<sup>29</sup> a soft gel could be formed as a consequence of loosening of non-covalent bonds due to disentanglement of amylopectin chains, which results in severe weakening of the remaining granule structure and therefore in softer starch granules. In addition, starch granules can also become soft due to breakage of covalent bonds that can occur during prolonged heating under shear.

The temperature  $T_{\text{gel}}$  at which the maximum  $G'$  ( $G'_{\max}$ ) was reached varied among the yam species and within the cultivars in a species (Table 3). The low  $T_{\text{gel}}$  observed in *D. rotundata* varieties may be a result of their higher starch content (Table 1) compared to the other yam species. This agreed with the report of Keetels *et al.*<sup>29</sup> that the higher the starch concentration, the lower the temperature at which a maximum  $G'$  can be reached during heating of the starch system. The correlation of this  $T_{\text{gel}}$  and the PT (viscoamylograph property) of the yam starches can imply that it is equivalent to the temperature at which the irreversible transition of starch during the gelatinization occurs. This temperature ( $T_{\text{gel}}$ ) can thus be considered an index of their PTs during the irreversible transition toward gelatinization in the rheological profile of raw yam tubers. This may also indicate that *D. rotundata* will have a shorter cooking time than the other yam species. In addition, it was also observed that among *D. rotundata* and *D. alata* varieties, the higher the  $G_{\max}$ , the lower was the  $T_{\text{gel}}$ . Thus,  $T_{\text{gel}}$  in raw yam tubers can be a useful screening tool for PT for potential utilization of starches from these yam tubers.

SC of the raw yam tubers (from its rheological profile) represents the point of swelling of the starch granule, crystalline melting, solubilization and breaking of intramolecular hydrogen bonds. The correlation between HPV of the starch (viscosity related to maximum granule swelling at plateau in the viscoamylograph characteristics) and the SC of the tubers depicted in Fig. 2(a) implied that the SC of the fresh yam tubers might be effectively related to the phenomenon of granule swelling during gelatinization. Hence, SC in the rheological profile of the fresh yam tubers is an index of the SC of its yam starch. The SC was different for the yam varieties. Differences in the SCs between the tubers can be added to differences in their starch content and also in the amount of structural entanglements (e.g., lipids, phosphate groups, non-starch carbohydrates, etc.). According to Steenekens<sup>31</sup> and Svegmarm and Hermansson<sup>32</sup> presence of entanglements in a starch system may lead to greater stiffness and reduce the sensitivity of the swollen granules to shear by increasing their resistance to disruption. The time taken for the starch granule to swell is represented by TS in the rheological profile; this is similar to the cooking ability

reported by Gibert *et al.*<sup>33</sup> on viscoamylograph of different varieties of banana, where the cooking ability was defined as the time taken for starch granule to attain full-swelling from its PT. The time taken for the starch gel in the raw yam tubers to rupture was depicted as TP. The time taken for the starch gels to rupture may be taken as an index of their resistance to mechanical shear. The tubers of *D. bulbifera* varieties did not rupture or lose their structural integrity easily (their  $T_{\text{gel}}$  was the highest; 81.6–84.3 °C). This may be a result of their strength and rigidity and may explain why *D. bulbifera* tubers had high initial  $G'$  and also why it took a long time for their gel to rupture compared to other species (Fig. 3(c)). Among the *D. bulbifera* species, TDb 3884 had the highest initial  $G'$  (about 6300 Pa) indicating the rigidity of its tuber. In addition, the rheological profile of raw tuber of this variety (Fig. 4(d)) did not show a distinct and clear gelatinization profile. This may be as a result of its lower starch content (Table 1) and low SC compared with the other yam species, hence exhibiting a more restricted swelling. This was similar to what was reported of the profile of waxy potato cultivar (Nadine) by Singh *et al.*<sup>12</sup> Furthermore in this study, *D. rotundata*, which had been reported<sup>27</sup> to produce mealy boiled yam, had high maximum storage modulus ( $G'_{\max}$ ) dry matter and starch contents as was reported for mealy potatoes by Singh *et al.*<sup>12</sup> Therefore, the rheological characteristics or profile of raw *D. bulbifera* tuber may be used as a template to predict waxiness in boiled yam tubers. High initial  $G'$  in the raw yam tubers may also indicate a tuber with low starch content and low SC.

From the rheographs of the raw tubers (Fig. 4(a)–(d)), the higher  $G'$  of the yam tubers than  $G''$  ( $G' > G''$ ) is an indication that their starches will be elastic or stretchable. This may indicate that the food products from varieties with higher  $G'$  may be more stretchable. In addition, food products from the yam varieties with higher  $G'$  may be more elastic or stretchable, thus influencing their food quality attributes. Hence, this can be used to predict the stretchability of yam food products such as pounded yam. The dynamic oscillatory characteristics of raw tubers can therefore be used to predict textural attributes (such as stretchability) in the food products (pounded yam) of yam varieties.

Decrease in the  $\tan\delta$  could be the result of gelation of the starch in the tuber tissue, a consequence of short-term amylose retrogradation of the starch which leached from the granules.<sup>12</sup> It can also be due to the interaction between starch molecules remaining inside the granules. It is noteworthy to mention the presence of both  $G'$  and  $G''$  shape peaks (Figs 2(a) and 4(a)–(d)), that is related to the phenomenon occurring during the decrease in the damping factor at the cooling stage.

As earlier reported,<sup>34</sup> stress relaxation test can be applied in uniaxial texture measurement to characterize the viscoelastic behavior or its failure strength, or decay in stress, with time when a constant strain is applied. It was also generally observed that the higher the dry matter of the tuber, the higher was the force and the work done to rupture the tuber. According to Rosenthal<sup>35</sup> texture of a food depends on the mechanical, geometrical and surface attributes, which are in turn dependent on the chemical composition of the food. Thus, the dependence of the force and the work-to-rupture on the dry matter of the tuber may be a result of the chemical composition (starch, non-starch carbohydrates and other parietal compounds) of the tubers, especially starch since it represents about 85% of yam tuber composition. This may be the reason the force to rupture *D. rotundata* tubers were higher compared to that for other species. The presence of higher starch content may account for the rigidity and compactness of the tubers.

### Interaction between dry matter, relaxation and rheological properties of the tuber

From Tables 1, 3 and 4, we observed some correlations between the composition, textural characteristics and rheological properties of the tubers: (a) the higher the dry matter of the tuber, the shorter was the time taken for the starch in the tuber to swell ( $r = -0.73$ ) and the shorter the time it took for the gel to rupture; (b) the higher the SC of the tuber, the shorter the time it took for the gel to rupture ( $r = -0.82$ ); (c) the lower the work done and the force to rupture the tuber ( $r = 0.87$ ), the lower was the SC ( $r = 0.54$  and  $r = 0.57$ ), respectively, and the higher the  $G'$  of the yam tubers; (d) the lower the SC, the higher the initial  $G'$  as observed in *D. bulbifera*.

In summary, this study established that dynamic rheological characterization of yam tubers can give information about the mechanical structure (rigidity and compactness of the tuber) and how this structure can affect the behavior of the tuber during processing, such as resistance to mechanical shear, swelling power, gelatinization, temperature and cooking time; thus, circumventing having to extract the starch from the tubers in order to determine these physicochemical characteristics. Hence it can serve as a MTPP method to screen for these parameters and predict the quality of yam food products. Characterizing the *in situ* rheological profile of the raw yam tuber samples rather than the starch gels can also serve as a rapid screening test for evaluating the physicochemical properties of the yam starch directly within a non-degraded matrix representing the tuber parenchyma structure, circumventing the drudgery of starch extraction. The  $T_{gel}$  of the yam tubers can be an index of the gelatinization temperature and PT of their starches, or cooking time, the SC of the tubers can be an index of the SC and PV of its yam starch without extracting starch from the tubers, thus a useful selection indicator for breeders. Rheological characterization of raw yam tubers can also be used as a tool to predict the properties of yam products, such as viscosity and elasticity of starch gel or stretchability of doughy yam food products in relation to yam tuber composition. The results also highlighted the distinctive structure of the four yam species especially *D. bulbifera* species. High initial  $G'$  as it occurred in *D. bulbifera*, may indicate a tuber with strong and rigid structure, which does not lose its structural integrity easily on heating, hence a higher  $T_{gel}$ . The study also showed that the higher the starch content the higher the  $G'_{max}$  and the shorter the time it takes to reach the  $T_{gel}$ . Time taken for starch gels to rupture can be taken as an index of their resistance to mechanical shear. SC can indicate the SC of the starch in the tubers. The stress relaxation test was also revealed as a relevant tool for *in situ* characterization of the work to rupture the tuber, being positively related to tuber dry matter content.

### AUTHOR CONTRIBUTIONS

Conceptualization: Bolanle Otegbayo and Olivier Gibert. Data curation: Bolanle Otegbayo and Olivier Gibert. Formal analysis: Bolanle Otegbayo and Olivier Gibert. Funding acquisition: Bolanle Otegbayo. Investigation: Bolanle Otegbayo and Olivier Gibert, Methodology: Olivier Gibert, Bolanle Otegbayo, Thierry Tran and Julien Ricci. Project administration: Olivier Gibert. Supervision: Olivier Gibert. Writing – original draft: Bolanle Otegbayo. Writing – review and editing: Bolanle Otegbayo and Olivier Gibert.

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### DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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