Research Article

Physicochemical Composition and Antioxidant Activity of Five Gari Processed from Cassava Roots (*Manihot esculenta Crantz*) Harvested at Two Different Maturity Stages and Two Seasons

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Gari is a partially gelatinized roasted fermented granular white or yellowish product made from storage roots of cassava. It is consumed as fast foods in many countries across the world. Physicochemical composition, particle size, colour, and antioxidant activities of five gari (92/0326, 96/1414, IRAD4115, EN, and AD) processed from fresh storage roots harvested at 12 months after planting (MAP) and 15MAP compared to four (4) commercial gari (M1, M2, M3, and M4) were evaluated. The analytical results revealed that colour value $b^*$ and particle size varied significantly ($p<0.05$) among the gari samples. Bound flavonoid contents were lower than free flavonoids (3.93 to 10.50 mgQE/100 g and 2.40 to 8.85 mgQE/100 g, respectively). Fourier transform infrared confirmed the functional groups in all gari samples. The antioxidant activity of the bound phenolics showed significantly ($p<0.05$) higher DPPH scavenging ability than free phenolics (gari M2: 2.70 μgTE/g). Similarly, the bound phenolics showed significant ($p<0.05$) variation of HRSA scavenging activity (0.18-35.09 μgTE/g). However, the best HRSA scavenging activity was found with bound phenolics of gari 96/1414, whereas HRSA scavenging activity was not detected in gari 92/0326, 96/1414, and AD. The value of ABTS scavenging activity of gari varied significantly ($p<0.05$) from 20.60 to 30.17 μgTE/g and from 20.70 to 34.39 for free and bound phenolics, respectively, while free phenolics showed higher FRAP value (7.97 mgTE/g) than the bound phenolics (4.59 mgTE/g). Additionally, phenolics and antioxidant activities showed significantly ($p<0.05$) a positive correlation. The present study has provided an insight into the physicochemical composition, bioactive compounds, and antioxidant activities of various gari processed at different season and maturity period of harvesting. It reveals that consumers of cassava gari can get health benefits apart from the nutritional values.

1. Introduction

Cassava (*Manihot esculenta Crantz*), which belongs to the family Euphorbiaceae is cultivated through the tropics. It is known as a staple food in many parts of the world, mostly in the developing countries. Their storage roots provided carbohydrates for more than 2 billion people in the tropics [1]. It serves also as an industrial raw material in pharmaceutical and chemical industries. Cassava leaves are consumed as green vegetable in various forms as well as used for medicinal purposes [2, 3]. It is also a source for biofuel as well as animal feed. In Cameroon, cassava was among the staple food throughout the country with the exception of the Northern Region. The leaves of cassava are used to prepare various sauce or soup and magani [4]. In general, cassava storage roots may spoil quickly after harvest (two to three days), so it is recommended to process into products such gari, semolina, flour, fufu, tapioca, and starch. These products were traditional foods that are consumed widely across the country. Among these by-products, gari, a fermented product, appears to be mostly appreciated for consumption due to its nutritional quality and relative long shelf life comparable to other processed products of cassava storage roots [1, 5]. Processing cassava storage roots into gari usually involves several processing steps due to the need to remove the toxic, cyanoglucoside contents, inherent to the
storage roots for its safe level. In addition, gari, a fermented
granulated food, had prebiotic and probiotic beneficial activ-
ity in the human body [6–8]. In recent decades, natural die-
tary antioxidants had gained much attention due to their
importance to protect body human against noncommunic-
able diseases such as obesity, diabetes, cancer, and hyperten-
sion [3, 9–11]. Among these compounds, phenolic is known
to be the most prominent natural dietary antioxidants
widely distributed in cassava plants. However, the quality
of the phenolic compounds may be altered during process-
ing of raw material to process some nutritional products
by using grinding, heating, sieving, etc. Hence, their bioac-
tivity decreased and affected directly the antioxidant activi-
ties due to the substantial losses of antioxidant components
[12]. In addition, the distribution of particle size in food
processed may be also a critical parameter for organoleptic
quality of the products. However, fermentation by microor-
ganisms could lead to the structural breakdown of cell walls,
leading to the synthesis of new metabolites and liberation of
various other bioactive ingredients with more bioactivity.
The functional properties and nutritional quality of gari pro-
duced at different periods compared to commercial gari have
been studied by Laya et al. [1]; however, an evaluation of
antioxidant activity of different fractions of phenolics from
this gari has been not carried out.

Thus, to our best knowledge, studies composed of total
free and bound phenolic compounds in cassava gari were
not reported. Nowadays, there was no information about
the antioxidant activity of cassava gari related to their phe-
nolic compounds, especially the free and bound phenolic
contents. Thus, assessed polyphenolics and their antioxidant
activities were important in order to know whether con-
sumers of cassava gari could get health benefits to improve
their well-being.

Therefore, the present study is aimed at assessing the
physicochemical, bioactive compounds, and antioxidant
properties of the processed cassava gari at different times
of the harvest compared to local commercial gari collected
in the local markets in order to know whether gari samples
processed from different varieties at different maturity
periods and seasons could be reliable as a source of nutri-
tional value with a high organoleptic property, antioxidant
activities were important in order to know whether con-
sumers of cassava gari were improved varieties, EN was local sweet variety, and AD
was also local bitter variety obtained from experimental field in
Tokombéré subdivision (10° 54’0.0” north latitude and
14° 12’0.0” east longitude), Maroua, Cameroon. Their stor-
age roots were harvested in dry season at twelve months
after planting (MAP) and in rainy season at fifteen MAP,
respectively.

2.3. Processing of Cassava Storage Roots into Gari. The cas-
sva storage roots were processed into gari using modified
method described by Agbor-Egbe and Mbome [13] and
Amamgbo et al. [14].

2.4. Colour Measurement. The colour of gari sample was
determined using the L∗ a∗ b∗ colour notation system with
spectrophotometer Konica Minolta (CM-5, Tokyo, Japan).
Forty (40) grams of sample was weighed into a dish, and
the colour was measured. The chromometer was calibrated
with a standard white tile. The instrument was set at illumi-
nant D65. Colour measurement was replicated three times.

2.5. Scanning Electron Microscopy (SEM). Samples of gari
were prepared as intact specimens to observe the structure
surface. The gari samples were mounted onto a stub using
conductive tape and sputter coated for 90 s with gold/palla-
dium. All samples were viewed in a Leo Electron Microscope
(model Leo 435VP, 1995; Cambridge, UK) scanning electron
microscope.

2.6. Particle Size. Fifty (50) grams of gari was sieved through
a set of graded Tyler sieves of aperture sizes 700, 500, 300,
250, 150, 100, 60, 40, 20, and 10 μm using a Microtrac (ana-
lysis mode: S3000/3500, Germany) set zero time 30 seconds
and run time 10 seconds for the total number of runs 3.
Fractions retained on each sieve were then weighed and used
to calculate the particle size distribution percent of the gari
samples.

2.7. FTIR Spectroscopy Analysis. Fourier transform infrared
(FTIR) spectrophotometer was used to analyze the func-
tional groups of the active components present in methano-
lic extract based on the peak values in the region of IR
radiation. Methanolic extracts (10 μL) of gari sample were
characterized using Fourier transform infrared spectrometer
Tensor II with platinum ATR (Tensor II, FTIR-Bruker,
Ettlingen, Germany) for FTIR spectrum measurement, 16
scans with the resolution of 4 in the frequency range of
400 to 4000 cm⁻¹. The instrumental control was carried
out using OPUS software (OPUS v. 7.0 for Microsoft, Bruker
Optics, Ettlingen, Germany).

2.8. Polyphenolic Extraction. Free and bound phenolic com-
 pounds were extracted following the method of Chen et al.
[15] with some changes. Shortly, 20 mL of methanol was
added into the fine ground gari powder (300 mg) in triplicate
for each analysis and mixed overnight by placing over danc-
ing shaker at room temperature (25°C). The mixture was
centrifuged at 10,000 rpm for 20 min at 4°C. The extraction
solution was filtrated with Whatman no. 1. Then, the residue

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was washed two times with 5 mL of methanol HPLC grade and then centrifuged. The supernatants were combined and centrifuged at the same conditions described above and filtered with Whatman no. 1. The solution was evaporated at 45°C to dryness and dissolved in methanol (HPLC grade) before keeping at -20°C until analysis. For bound phenolic content, the residue resulting from free phenolic extraction was mixed with 10 mL NaOH (2N) solution and boiled in shaking water bath at 50 rpm for 30 min before adding 5 mL HCl (2 M), and it was incubated for 60 min at 60°C. The extraction mixture was cooled, and 10 mL of ethyl acetate was added into the solution. After 1 h shaking, the solution was filtered and the ethyl acetate fraction was centrifuged as described above. The residue obtained after filtration was mixed more time with 10 mL of ethyl acetate and centrifuged at the same conditions as described above before filtering through Whatman no. 1. The combined supernatant was evaporated at 45°C to dryness using rota vapor. The residue obtained was dissolved in methanol HPLC grade and centrifuged as described previously. The methanol extract was kept at -20°C until analysis.

2.9. Flavonoid Content. Free and bound flavonoids contents were determined as described by Xiong et al. [16] with some modifications using multimode reader (Tecnac SPARK 10M, V1.2.20, Austria). In brief, 20 μL of sample was mixed with 20 μL of 5% sodium nitrite and of 2% aluminum chloride. Then, the mixture was incubated for 3 min before adding 50 μL NaOH (0.5 M). The solution mixture was mixed again for 40 s and incubated for 6 min. Then, 30 μL of 2% sodium acetate was added in each well and incubated for 40 min in the dark place. The absorbance was recorded at 510 nm using multimode reader (Tecnac SPARK 10M, V1.2.20, Austria). The quercetin was used as a standard.

2.10. Total Tannin Content. Total tannin content was determined using the vanillin reagent according to Gaytan-Martínez et al. [17] with some modifications using multimode reader. First, 20 μL of the sample was mixed with 120 μL of vanillin reagent (4% vanillin in methanol). The mixture was incubated for 3 min, and after that, 30 μL of concentrated HCl (36.5%) was added. The microplate was covered with aluminum foil and incubated for 20 min in the dark. The absorbance was taken at 500 nm in multimode reader (Tecnac SPARK 10M, V1.2.20). The tannic acid was used as a standard.

2.11. Antioxidant Activity Assay

2.11.1. Ferric Reducing Antioxidant Power (FRAP). The FRAP was carried out according to the method described by Feregrino-Pérez et al. [18] with minor changes. Using a micropipette, 20 μL of the sample or blank was added to 120 μL of the FRAP reagent in a 96-well microplate and the mixture was shaken for 50 s and then incubated for 40 min. The absorbance was recorded at 595 nm with multimode reader (Tecnac SPARK 10M, V1.2.20), and the results were determined using the Trolox as a standard.

2.11.2. 2,2-Diphenyl-1-Picrylhydrazyl (DPPH). The DPPH radical scavenging activity of the cassava gari was carried out according to the method described by Feregrino-Pérez et al. [18] with some modifications. To the sample solution (40 μL), 150 μL of DPPH (1 mM) was added in a 96-well microplate (New York, USA). The solution was mixed for 40 s using multimode reader (Tecnac SPARK 10M, V1.2.20, Austria) and incubated for 40 min in the dark place. After incubation, the absorbance was recorded at 517 nm with multimode reader (Tecnac SPARK 10M, V1.2.20). The results were evaluated from the Trolox as a standard.

2.11.3. 2,2-Azinobis-(3-Ethylbenzothiazoline-6-Sulfonic Acid) (ABTS). ABTS radical scavenging was performed as previously reported by Wu et al. [19] with some modifications using multimode microplate reader (Tecnac SPARK 10M, V1.2.20, Austria). The reaction started by adding 20 μL of the extract or blank into 150 μL of ABTS+ solution. The microplate was shaken for 50 s before incubation at room temperature for 40 min in the dark. The absorbance was taken at 734 nm, and the results were evaluated from the Trolox as a standard.

2.11.4. Hydroxyl Radical Scavenging Activity (HRSA). HRSA was evaluated as previously described by Feregrino-Pérez et al. [20] with some modifications. Shortly, 50 μL of FeSO4 (9 mM), 50 μL of H2O2 (0.03%, v/v), 50 μL of salicylic acid-ethanol solution (9 mM), and 50 μL of extract were mixed in 96-well microplate (New York, USA) and incubated for 40 min in the dark place. The absorbance was taken at 510 nm with multimode microplate reader. Trolox was used as a control. HRSA was calculated from the Trolox as a standard curve plotted with seven concentrations (5-1000 μg/mL).

2.12. Statistical Analysis. ANOVA was carried out using Statgraphics software (version 16). Tukey’s (HSD) test was used to determine any significant difference between different gari, and significance was accepted at level p < 0.05. Correlation and principal component analysis (PCA) were done to establish the relationship between antioxidant activities and phenolic compounds. The results were expressed as means ± standard deviation. The experiments were carried out in four replications.

3. Results and Discussion

3.1. Physical Properties of Gari Samples

3.1.1. Colour. Colour is one important factor attribute that determines the quality of gari. It is also one of the key quality attribute that influence consumer’s behavior, choices, and perceptions [21]. The L* a* b* colour notation was used to describe the colour of the gari samples, where a* means the degree of redness or greenness and L* and b* indicate the degree of lightness and the intensity of yellowness, respectively [21]. The difference in the colour of the gari samples was very significant (p < 0.05) (Table 1). This difference observed in the colour of the gari samples obviously resulted from the fact that storage roots used for garification...
were harvested at different stages and seasons that affected the physiological changes. The gari produced from roots harvested at 12MAP and 15MAP had significantly affected the colour of gari, which was also greatly different from the commercial gari samples (Table 1). Thus, period of the harvest seems to have significantly (p < 0.05) affected the colour of the gari samples. The values b* of gari produced at 12MAP varied from 29.99 to 37.87 while those at 15MAP only on small-scale by Chuzel et al. [23] who recommended the range of 17-21. However, higher than that reported the present study was slightly similar as observed by Oduro et al. [22] in their work, however, higher than that reported by Hidayat et al. [24], hence starch breakdown in small size by Maroua (Far North Region) and Bertoua (East Region) (Cameroon).

Table 1: Colour of processed gari at 12MAP and at 15MAP compared to four (04) commercial gari collected in the local markets, Abattoir Maroua (Far North Region) and Bertoua (East Region) (Cameroon).

<table>
<thead>
<tr>
<th>Harvest times</th>
<th>Gari</th>
<th>L*</th>
<th>a*</th>
<th>b*</th>
</tr>
</thead>
<tbody>
<tr>
<td>12MAP</td>
<td>TMS92</td>
<td>69.06 ± 0.28d</td>
<td>8.77 ± 0.24d</td>
<td>37.87 ± 0.56c</td>
</tr>
<tr>
<td>15MAP</td>
<td>M1</td>
<td>69.88 ± 0.40d</td>
<td>6.62 ± 0.49b</td>
<td>28.90 ± 0.76b</td>
</tr>
<tr>
<td>12MAP</td>
<td>TMS96</td>
<td>69.90 ± 0.93d</td>
<td>7.43 ± 0.53c</td>
<td>29.99 ± 0.57c</td>
</tr>
<tr>
<td>15MAP</td>
<td>M2</td>
<td>73.94 ± 0.30b</td>
<td>5.01 ± 0.04d</td>
<td>25.93 ± 0.15b</td>
</tr>
<tr>
<td>12MAP</td>
<td>IRAD</td>
<td>68.74 ± 0.42de</td>
<td>7.89 ± 0.20f</td>
<td>33.18 ± 0.79e</td>
</tr>
<tr>
<td>15MAP</td>
<td>M3</td>
<td>78.29 ± 0.57a</td>
<td>3.67 ± 0.16b</td>
<td>25.39 ± 0.41bi</td>
</tr>
<tr>
<td>12MAP</td>
<td>EN</td>
<td>74.01 ± 0.07b</td>
<td>5.45 ± 0.13e</td>
<td>25.49 ± 0.35i</td>
</tr>
<tr>
<td>15MAP</td>
<td>M4</td>
<td>67.70 ± 0.34c</td>
<td>8.74 ± 0.21d</td>
<td>31.64 ± 0.09f</td>
</tr>
<tr>
<td>/</td>
<td>M1</td>
<td>58.08 ± 0.07e</td>
<td>9.14 ± 0.43e</td>
<td>29.06 ± 0.45e</td>
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<tr>
<td>/</td>
<td>M2</td>
<td>68.39 ± 0.13e</td>
<td>11.12 ± 0.15f</td>
<td>48.56 ± 0.69a</td>
</tr>
<tr>
<td>/</td>
<td>M3</td>
<td>70.57 ± 0.21c</td>
<td>6.79 ± 0.21f</td>
<td>36.44 ± 0.73f</td>
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<tr>
<td>/</td>
<td>M4</td>
<td>67.05 ± 0.82e</td>
<td>11.74 ± 0.21f</td>
<td>48.35 ± 0.52b</td>
</tr>
<tr>
<td>/</td>
<td>M4</td>
<td>70.46 ± 0.24c</td>
<td>4.20 ± 0.27f</td>
<td>26.17 ± 0.68b</td>
</tr>
</tbody>
</table>

12MAP: 12 months after planting (dry season); 15MAP: 15 months after planting (raining season); /: unknown months of the harvests. M1, M2, M3, and M4 were commercial gari collected from Abattoir Maroua and Bertoua markets, respectively. Values followed by different letters in each column are different (p < 0.05) significantly.

were commercial gari collected from Abattoir Maroua and Bertoua markets, respectively. Values followed by different letters in each column are different (p < 0.05) significantly.

3.1.2. Scanning Electron Microscopy (SEM). Starch granule damage morphology of gari samples was studied using a SEM. Starch granule damage morphology from gari produced at 12MAP had a different morphology (Figure 1(a)) compared to those gari produced at 15MAP (Figure 1(b)) as well as commercial gari (Figure 1(c)). The starch granules of gari samples had broken with fissures, microholes, and rough surfaces, while some granules were clumped together and showed a cluster with large aggregates (Figure 1(b)). This result suggests that fiber matrix structure was damaged during fermentation by microorganism activity as reported by Hidayat et al. [24], hence starch breakdown in small size granules. On the other hand, the modified starch may have high amylose content; thus, excessive leaching of this amylose causes the starch granules to collapse resulting in gelatinization [25]. This indicates more severe damage of starch granules during fermentation and roasting process. The modification of starch morphology may improve the functionality of gari including water absorption, solubility, and swelling power. However, the clumped granules may reduce the water absorption capacity and solubility of the gari samples. It was reported that the changes of structure and morphology of starch granules impacted deeply on the pasting properties and thus increased in starch digestibility due to damaged granules [26]. Furthermore, the smaller granules of gari may expand to a great surface area and better digestion due to contact of food with enzyme activity [27]. It is obvious that the damage of starch granules depends on the type of gari as affected by varietal differences (Figure 1).

3.1.3. Particle Size Distribution. Apart from the appearance as among the factors which affect the quality of gari, particle size seems to be one of the most important for consumers [28]. On the other hand, in nutrition, particle size has significant effect on food digestibility in the gastrointestinal system [29]. Thus, study distribution of particle size of ready food for consumer is a challenge for the food industry in order to offer a high organoleptic quality. In this study, the results of particle size of gari samples are shown in Table 2. On mesh sizes 10 μm and 20 μm, the percentage retention varies from 100 to 99.39 and 100 to 90.97%, respectively. Mesh sizes 40 and 60 μm have percentage retention varying from 100 to 79.9.66% and 100 to 63.41%, respectively. However, on mesh sizes 100 and 150 μm, the percentage retention varies from 100 to 52.62 and 100 to 52.33%, respectively. On mesh sizes 250 and 300 μm, the percentage retention varies from 99.60 to 42.69 and 97.36 to 30.76%, respectively. While on mesh sizes 500 and 700 μm, the percentage retention varies from 84.35 to 36.25 and 77.51 to 5.75%, respectively (Table 2). Variation in particle size distribution of gari sample size may be due...
Figure 1: Continued.
Figure 1: Continued.
to the differences in the grating procedure, roasting process, and the period of fermentation of the cassava mash compared to commercial *gari*. In fact, fermentation has been shown to affect particle size [22]. The variation in particle size may also be attributed to the stage of harvest cassava storage roots to process into *gari* and variety as well. The appearance is one factor that dictates the quality of *gari*; however, the particle size distribution is the most important

**Figure 1:** SEM of different gari samples: (a) produced gari at 12MAP (dry season); (b) processed gari at 15MAP (rainy season); (c) commercial gari.

**Table 2:** Particle size distribution of processed gari compared to four (04) commercial gari as reference collected in the local markets, *Abattoir Maroua* (Far North Region) and *Bertoua* (East Region) (Cameroon).

<table>
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<th>Harvest times</th>
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<th>10</th>
<th>20</th>
<th>40</th>
<th>60</th>
<th>100</th>
<th>150</th>
<th>250</th>
<th>300</th>
<th>500</th>
<th>700</th>
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<td>100.00</td>
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<td>100.00</td>
<td>100.00</td>
<td>100.00</td>
<td>99.60</td>
<td>97.36</td>
<td>84.35</td>
<td>77.51</td>
<td></td>
</tr>
</tbody>
</table>

12MAP: 12 months after planting (dry season); 15MAP: 15 months after planting (rainy season); /: unknown months of the harvest. M1, M2, M3, and M4 were commercial gari collected from *Abattoir Maroua* and *Bertoua* markets, respectively.
for consumers because they will most likely have high preference for fine particles [30]. Furthermore, the separation of particles on the basis of particle size distribution may help to provide some information on functional properties of products processed including water hydration and textural property [29].

3.2. FTIR Spectroscopy. Fourier transform infrared (FTIR) is the most powerful tool for identifying the types of functional groups (chemical bonds) present in gari. The FTIR spectra related to studied samples are shown in Figure 2. The bands identified in samples displayed in Table 3 revealed the presence of many functional groups in methanolic extracts of gari. The FTIR spectrum confirmed the presence of phenols, amino acids, amides, carboxylic acids, alkanes, aliphatic esters, secondary alcohols, sulfur compounds, and mono substituted alkenes in methanolic extract of gari samples. The functionality of gari including water absorption and solubility may be influenced by proteins, amino acids, and other functional groups of the gari revealed by FTIR (Table 3). On the basis of FTIR spectra, there was partial gelatinization that occurred disrupting the ordered crystalline structure of amylopectin of starch [25]. Finally, possible leaching of amylose resulting from heat treatment increases the chances of the different molecules interacting with each other including hydrogen bonding and amylose [25], and this interaction may improve the swelling power of gari which is the one parameter influencing the gari consumption by consumers.

![Figure 2: FTIR spectra of methanolic extracts of all gari samples. G1512: gari 4115 produced at 12MAP (months after plantation, dry season); G1515: gari 4115 produced at 15MAP (raining season); G9212: gari 92/0326 produced at 12MAP; G9215: gari 92/0326 produced at 15MAP; G9612: gari 96/1414 produced at 12MAP; G9615: gari 96/1414 produced at 15MAP; GAD12: gari AD produced at 12MAP; GAD15: gari AD produced at 15MAP; GEN12: gari EN produced at 12MAP; GEN15: gari EN produced at 15MAP; M1, M2, M3, and M4: commercial gari.](image)

<table>
<thead>
<tr>
<th>Peak values</th>
<th>Functional groups</th>
<th>Peak values</th>
<th>Functional groups</th>
</tr>
</thead>
<tbody>
<tr>
<td>3318-3436</td>
<td>Phenol</td>
<td>1648-1745</td>
<td>Aliphatic</td>
</tr>
<tr>
<td>3300-3696</td>
<td>Amine</td>
<td>1648</td>
<td>Amide</td>
</tr>
<tr>
<td>3000-3050</td>
<td>Aromatic</td>
<td>974</td>
<td>Alkene</td>
</tr>
<tr>
<td>3317</td>
<td>Amide</td>
<td>1415-1450</td>
<td>Aromatic</td>
</tr>
<tr>
<td>2832-2943</td>
<td>Aliphatic</td>
<td>1231-1283</td>
<td>Ether</td>
</tr>
<tr>
<td>2527-2693</td>
<td>Acid</td>
<td>1115</td>
<td>Secondary alcohol</td>
</tr>
<tr>
<td>2171-223</td>
<td>Amino acid</td>
<td>1020-1033</td>
<td>Sulfur</td>
</tr>
</tbody>
</table>

3.3. Polyphenolic Compounds of Methanolic Extracts of Gari Samples

3.3.1. Flavonoid Contents. As shown in Table 4, there was significant \((p < 0.05)\) variation of free and bound flavonoid content in gari samples. However, the highest value (10.50 mgQE/100 g) of free flavonoids was shown by commercial gari M1, while the lowest value (3.93 mgQE/100 g) was observed in gari AD produced at 15MAP (Table 4). These results suggest that a variety may affect the flavonoid levels in gari samples. The highest value of free flavonoids could be attributed to variety and season of the harvest periods of storage roots used for garification [31]. Variation of flavonoid contents in gari samples could be attributed to processing methods as shown by commercial gari. The lowest value (2.42 mgQE/100 g) of bound flavonoid was shown by
Table 4: Polyphenolic compounds (free and bound) of methanolic extracts of processed gari compared to four (04) commercial gari as reference collected in the local markets, Abattoir Maroua (Far North Region) and Bertoua (East Region) (Cameroon).

<table>
<thead>
<tr>
<th>Harvest times</th>
<th>Gari</th>
<th>Free flavonoids (mgQE/100 gdw)</th>
<th>Bound flavonoids (mgQE/100 gdw)</th>
<th>Free tannins (mgTAE/100 gdw)</th>
<th>Bound tannins (mgTAE/100 gdw)</th>
</tr>
</thead>
<tbody>
<tr>
<td>12MAP</td>
<td>TMS92</td>
<td>8.85 ± 0.49&lt;sup&gt;a&lt;/sup&gt;</td>
<td>5.48 ± 0.12&lt;sup&gt;d&lt;/sup&gt;</td>
<td>2.48 ± 0.03&lt;sup&gt;e&lt;/sup&gt;</td>
<td>1.19 ± 0.07&lt;sup&gt;cd&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>TMS96</td>
<td>8.37 ± 0.68&lt;sup&gt;f&lt;/sup&gt;</td>
<td>8.64 ± 0.02&lt;sup&gt;e&lt;/sup&gt;</td>
<td>4.28 ± 0.46&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.89 ± 0.01&lt;sup&gt;c&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>4114</td>
<td>10.16 ± 1.44&lt;sup&gt;ab&lt;/sup&gt;</td>
<td>3.82 ± 0.48&lt;sup&gt;g&lt;/sup&gt;</td>
<td>3.22 ± 0.14&lt;sup&gt;cd&lt;/sup&gt;</td>
<td>1.38 ± 0.01&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>EN</td>
<td>8.23 ± 0.52&lt;sup&gt;f&lt;/sup&gt;</td>
<td>2.42 ± 0.22&lt;sup&gt;d&lt;/sup&gt;</td>
<td>2.41 ± 0.15&lt;sup&gt;e&lt;/sup&gt;</td>
<td>0.92 ± 0.02&lt;sup&gt;de&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>AD</td>
<td>6.62 ± 0.40&lt;sup&gt;g&lt;/sup&gt;</td>
<td>7.89 ± 0.79&lt;sup&gt;c&lt;/sup&gt;</td>
<td>3.57 ± 0.12&lt;sup&gt;c&lt;/sup&gt;</td>
<td>0.67 ± 0.02&lt;sup&gt;e&lt;/sup&gt;</td>
</tr>
<tr>
<td>15MAP</td>
<td>TMS92</td>
<td>8.59 ± 0.64&lt;sup&gt;c&lt;/sup&gt;</td>
<td>8.85 ± 1.87&lt;sup&gt;a&lt;/sup&gt;</td>
<td>4.42 ± 0.21&lt;sup&gt;ab&lt;/sup&gt;</td>
<td>0.69 ± 0.08&lt;sup&gt;f&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>TMS96</td>
<td>6.38 ± 0.05&lt;sup&gt;b&lt;/sup&gt;</td>
<td>2.64 ± 0.58&lt;sup&gt;g&lt;/sup&gt;</td>
<td>2.19 ± 0.04&lt;sup&gt;f&lt;/sup&gt;</td>
<td>0.67 ± 0.02&lt;sup&gt;e&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>4114</td>
<td>9.70 ± 0.63&lt;sup&gt;c&lt;/sup&gt;</td>
<td>2.40 ± 0.26&lt;sup&gt;i&lt;/sup&gt;</td>
<td>3.23 ± 0.02&lt;sup&gt;c&lt;/sup&gt;</td>
<td>0.49 ± 0.01&lt;sup&gt;g&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>EN</td>
<td>6.00 ± 0.58&lt;sup&gt;i&lt;/sup&gt;</td>
<td>8.69 ± 1.57&lt;sup&gt;b&lt;/sup&gt;</td>
<td>4.59 ± 0.12&lt;sup&gt;a&lt;/sup&gt;</td>
<td>1.09 ± 0.03&lt;sup&gt;d&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>AD</td>
<td>3.93 ± 0.41&lt;sup&gt;j&lt;/sup&gt;</td>
<td>4.01 ± 1.74&lt;sup&gt;e&lt;/sup&gt;</td>
<td>1.60 ± 0.04&lt;sup&gt;d&lt;/sup&gt;</td>
<td>1.25 ± 0.02&lt;sup&gt;ab&lt;/sup&gt;</td>
</tr>
<tr>
<td>/</td>
<td>M1</td>
<td>10.50 ± 0.14&lt;sup&gt;a&lt;/sup&gt;</td>
<td>3.25 ± 0.59&lt;sup&gt;h&lt;/sup&gt;</td>
<td>0.46 ± 0.00&lt;sup&gt;i&lt;/sup&gt;</td>
<td>0.21 ± 0.01&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>M2</td>
<td>5.12 ± 0.13&lt;sup&gt;j&lt;/sup&gt;</td>
<td>5.07 ± 0.08&lt;sup&gt;e&lt;/sup&gt;</td>
<td>1.19 ± 0.05&lt;sup&gt;h&lt;/sup&gt;</td>
<td>1.55 ± 0.01&lt;sup&gt;i&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>M3</td>
<td>4.63 ± 0.28&lt;sup&gt;c&lt;/sup&gt;</td>
<td>4.44 ± 0.06&lt;sup&gt;f&lt;/sup&gt;</td>
<td>0.40 ± 0.17&lt;sup&gt;e&lt;/sup&gt;</td>
<td>0.68 ± 0.03&lt;sup&gt;j&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>M4</td>
<td>10.22 ± 2.50&lt;sup&gt;a&lt;/sup&gt;</td>
<td>2.76 ± 0.01&lt;sup&gt;i&lt;/sup&gt;</td>
<td>0.68 ± 0.01&lt;sup&gt;i&lt;/sup&gt;</td>
<td>0.27 ± 0.01&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

12MAP: 12 months after planting (dry season); 15MAP: 15 months after planting (rainy season); QE: quercetin equivalent; TAE: tannic acid equivalent; dw: dry weight; /: unknown months of the harvests. M1, M2, M3, and M4 were commercial gari collected from Abattoir Maroua and Bertoua markets, respectively. Values followed by different letters in each column are different (p < 0.05) significantly.

3.3.2. Total Tannin Contents (TTC). The TTC varied significantly (p < 0.05) in gari samples (Table 4). For free tannin content, the highest value (4.59 mgTAE/100 g) was shown by gari EN produced at 12MAP whereas the commercial gari M1 showed the lowest concentration (0.4 mgTAE/100 g). The gari produced at 15MAP recorded the highest value of tannin than those produced at 12MAP that suggest the effect of harvesting stages. Both bound and free TTC are significantly higher in gari produced at 12MAP and 15MAP compared to commercial gari, suggesting variety and processing methods had significant (p < 0.05) effect. The tannin contents observed in this study were higher than those (0.02-0.05 mg/100 g) obtained by Oluba et al. [33]. However, they did not mention in their study whether the polyphenols of the samples were free or bound fraction. The values of tannins (0.18-0.19 g/100 g) observed by Rocchetti et al. [34] were higher than the values obtained in the present study. In addition, Airodion et al. [31] reported higher values (0.22-0.30 g/100 g) of tannins in two types of gari.

3.4. Antioxidant Activities

3.4.1. Ferric Reducing Antioxidant Power (FRAP). FRAP of free and bound extract gari samples is shown in Table 5 with values ranging from 2.78 to 7.97 mgTE/gdw and 0.78 to 4.59 mgTE/gdw, respectively. For free extracts, EN gari produced at 12MAP had significantly (p < 0.05) higher FRAP compared to the other gari samples, while AD gari extracts had the lowest FRAP value (2.78 mgTE/gdw). The 92/0326 gari extracts of bound fraction showed the highest FRAP value, while AD gari had the lowest FRAP value. These variations of FRAP values of free and bound fractions of polyphenols were reported by Rocchetti et al. [34] on cooked gluten-free pasta. However, FRAP activity was significantly higher in free fraction polyphenols compared to the bound fraction. Similar result was obtained by Laya and Koubala [2], who found that the free form of phenols in cassava leaves showed the highest FRAP activity than bound form of polyphenols. The results suggest that free polyphenols may have the ability to reduce the potassium ferricyanide (Fe<sup>3+</sup>) into potassium ferrocyanide (Fe<sup>2+</sup>) than the bound fraction of polyphenols. On the other hand, dry heating may improve the phenolic bioactivity, which helps to enhance the antioxidant activity [35].

3.4.2. Ferric Reducing Antioxidant Power (DPPH). DPPH radical scavenging capacity of free and bound extracts of gari samples is shown in Table 5 with values ranging from 3.76 to 13.05 μgTE/gdw and 2.70 to 22.01 μgTE/gdw, respectively. Free fraction of polyphenols of AD gari produced at 12MAP showed significantly (p < 0.05) the highest value of DPPH radical scavenging activity compared to the other gari samples tested while the EN gari extracts had the lowest value of DPPH radical scavenging capacity (3.76 μgTE/gdw). The bound fraction of polyphenolic extracts of commercial gari, M4, showed significantly (p < 0.05) the highest value of DPPH radical scavenging activity (22.01 μgTE/gdw) followed by EN gari (19.63 μgTE/gdw) produced at 15MAP, indicating the lowest antioxidant activity. Similar results were obtained on sweet corn in China and various breads by Zhang et al. [36] and Verardo et al. [37], respectively.
Table 5: Antioxidant properties of methanolic extracts (free and bound polyphenolics) of processed gari compared to four (04) commercial gari as reference collected in the local markets, Abattoir Maroua (Far North Region) and Bertoua (East Region) (Cameroon).

<table>
<thead>
<tr>
<th>Harvest times</th>
<th>Gari</th>
<th>FRAP (mg TE/gdw)</th>
<th>DPPH (μg TE/gdw)</th>
<th>ABTS (μg TE/gdw)</th>
<th>HRSA (μg TE/gdw)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Free</td>
<td>Bound</td>
<td>Free</td>
<td>Bound</td>
</tr>
<tr>
<td>12MAP</td>
<td>TMS92</td>
<td>5.95 ± 0.67</td>
<td>3.63 ± 0.21</td>
<td>11.69 ± 0.12</td>
<td>7.42 ± 0.29</td>
</tr>
<tr>
<td></td>
<td>TMS96</td>
<td>4.89 ± 0.23</td>
<td>4.32 ± 0.31</td>
<td>6.17 ± 0.30</td>
<td>4.15 ± 0.19</td>
</tr>
<tr>
<td></td>
<td>4115</td>
<td>6.53 ± 0.22</td>
<td>5.94 ± 0.24</td>
<td>11.36 ± 0.30</td>
<td>6.17 ± 0.30</td>
</tr>
<tr>
<td></td>
<td>4115</td>
<td>7.97 ± 0.17</td>
<td>2.52 ± 0.19</td>
<td>11.91 ± 0.63</td>
<td>8.16 ± 0.10</td>
</tr>
<tr>
<td></td>
<td>AD</td>
<td>5.37 ± 0.21</td>
<td>1.78 ± 0.03</td>
<td>13.05 ± 0.16</td>
<td>4.76 ± 0.01</td>
</tr>
<tr>
<td>15MAP</td>
<td>TMS92</td>
<td>5.71 ± 0.36</td>
<td>4.59 ± 0.23</td>
<td>12.01 ± 0.77</td>
<td>6.08 ± 0.13</td>
</tr>
<tr>
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<td>TMS96</td>
<td>3.78 ± 0.08</td>
<td>3.36 ± 0.26</td>
<td>4.94 ± 0.31</td>
<td>10.09 ± 1.45</td>
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<td>4115</td>
<td>3.83 ± 0.24</td>
<td>2.78 ± 0.24</td>
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<td>6.35 ± 0.28</td>
</tr>
<tr>
<td></td>
<td>M1</td>
<td>4.39 ± 0.12</td>
<td>3.12 ± 0.19</td>
<td>4.23 ± 0.20</td>
<td>3.05 ± 0.51</td>
</tr>
<tr>
<td></td>
<td>M2</td>
<td>4.01 ± 0.33</td>
<td>3.58 ± 0.03</td>
<td>10.04 ± 0.34</td>
<td>2.70 ± 0.14</td>
</tr>
<tr>
<td></td>
<td>M3</td>
<td>3.07 ± 0.14</td>
<td>2.95 ± 0.21</td>
<td>5.47 ± 0.23</td>
<td>4.87 ± 0.31</td>
</tr>
<tr>
<td></td>
<td>M4</td>
<td>4.67 ± 0.38</td>
<td>3.87 ± 0.53</td>
<td>12.37 ± 0.14</td>
<td>22.01 ± 0.44</td>
</tr>
</tbody>
</table>

12MAP: 12 months after planting (dry season); 15MAP: 15 months after planting (raining season); TE: Trolox equivalent; dw: dry weight; ND: not detected; /: unknown months of the harvests. M1, M2, M3, and M4 were commercial gari collected from Abattoir Maroua and Bertoua markets, respectively. Values followed by different letters in each column are different (p < 0.05) significantly.

3.4.3. 2,2-Azino-bis-(3-Ethylbenzothiazoline-6-Sulfonic Acid) (ABTS). The ABTS activity of free and bound phenolic extracts of gari samples is shown in Table 5 with values ranging from 20.60 to 30.17 μg TE/gdw and 20.70 to 34.39 μg TE/gdw, respectively. The free fraction of phenolic extracts of AD gari produced at 15MAP showed significantly (p < 0.05) higher value of ABTS activity compared to the other gari samples, while the commercial gari extracts M4 had the lowest value of ABTS capacity (20.60 μg TE/gdw), indicating the highest antioxidant activity. These results were in agreement with Aïraodion et al. [31], who found that the bound phenolic compounds of various bread had higher antioxidant activity than free fraction of phenolics. The bound fraction extracts of phenolics of AD gari produced at 15MAP showed significantly (p < 0.05) the highest value of ABTS capacity (34.39 μg TE/gdw) among the other gari samples while commercial gari M2 had a value of 20.70 μg TE/gdw. The present results were similar to the antioxidant activity of bound and free fractions of phenolic compounds in sorghum obtained by Wu et al. [19]. The results may be due to the liberation of new metabolites and various antioxidant ingredients during the breakdown of plant cell walls through fermentation by microorganisms, which helps to increase the antioxidant activity [38].

3.4.4. Hydroxyl Radical Scavenging Activity (HRSA). The HRSA activity of free and bound extracts of phenolics of gari samples ranges from 2.58 to 14.80 μg TE/gdw and 0.18 to 35.09 μg TE/gdw, respectively. However, the HRSA activity of free fraction of phenolics (gari from 92/0326 and 96/1414 at 6MAP and AD at 9MAP) was not detected (Table 2). These results may suggest that variety differences and harvesting time of storage roots used to produce gari affect the quality of phenolic compounds. The free fraction of phenolics of commercial gari M4 showed significantly (p < 0.05) higher HRSA activity compared to the other gari samples, while the 4115 gari extracts produced at 12MAP had the lowest value of HRSA scavenging capacity (2.58 μg TE/gdw). The bound fraction extracts of 96/1414 gari produced at 15MAP showed significantly (p < 0.05) the highest value of HRSA value (35.09 μg TE/gdw) compared to the other gari samples, while commercial gari M3 had the lowest value (0.18 μg TE/gdw), indicating the highest antioxidant activity. This result was in agreement with Laya and Koubala [2], who found that the bound fraction of phenolics in cassava leaves had higher antioxidant activity than free fraction of phenolics, and this activity varied significantly (p < 0.05) among gari. Similarly, Liyana-Pathiraka and Shahidi [39] reported that bound form of phenolic contents had higher antioxidant activity than free phenolic compounds in wheat. It was also reported that fermentation is effective at increasing phenolics and antioxidant activity [40–42].

Furthermore, there are many other parameters which imply in antioxidant activity; for example, the heated or processed products may increase the activity including the Maillard reaction or caramelization reactions which occur during heating involve reducing sugars reacting with amino acids, peptides, or proteins producing products that have potential antioxidant properties (including FRAP, DPPH, and ABTS) which may improve the gastrointestinal health [43–45]. These products act by many mechanisms, including metal chelation and free radical scavenging [44]. The Maillard reaction products are capable of binding metal ions and scavenging free radicals and can thereby improve the oxidative stability of lipids [46]. Additionally, many researchers reported also the effects of particle size on the bioactive phenolics and antioxidant activity. For example, Prasedy et al. [47] reported significantly stronger...
antioxidant activity including FRAP, ABTS, and DPPH in macroalgae samples with smaller particle sizes than coarser particle distribution. Similarly, Dziki et al. [48] found that the antioxidant activity of oat husk (by-product from oat processing) increased as the particle size of the micronized husk decreased. Overall, gari samples EN and M1 with smaller particle size distribution than others showed low to moderate antioxidant activity with some discrepancies which may be due to varietal effects. The results may be also different because bioactive phenolics were extracted in different fractions and the antioxidant activity was assessed in the form of free and bound which may affect deeply the present results. The results suggested that the matrix of sample and types of samples affect the bioactive compounds and consequently their antioxidant activities. The results are in agreement with Nabil et al. [49] who found that bioactive compounds and antioxidant activity (FRAP, DPPH, and ABTS) of cladode powder were differentially distributed according to particle size with some fractions having high antioxidant activity.

3.5. Correlation between Antioxidant Activity, Flavonoid, and Tannin Content. Table 6 shows the results of different correlations between antioxidant activity of bound and free phenolics and polyphenolic compounds (bound and free). High positive correlation ($r = 0.638$, $p < 0.05$) was found between FRAPf and FRAPb assays. Similarly, there was a significantly positive relationship ($p < 0.05$) between HSRAf

<table>
<thead>
<tr>
<th>Variables (PC1 and PC2: 46.21 %)</th>
<th>Flaf</th>
<th>Tanf</th>
<th>ABTSf</th>
<th>DPPHf</th>
<th>FRAPf</th>
<th>HSRAf</th>
<th>Flab</th>
<th>Tanb</th>
<th>DPPHb</th>
<th>FRAPb</th>
<th>HSRAb</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flaf</td>
<td>1</td>
<td>0.065</td>
<td>-0.091</td>
<td>0.311</td>
<td>0.27</td>
<td>0.15</td>
<td>-0.102</td>
<td>0.282</td>
<td>-0.179</td>
<td>-0.035</td>
<td>0.45</td>
</tr>
<tr>
<td>Tanf</td>
<td>1</td>
<td>0.213</td>
<td>0.04</td>
<td>-0.209</td>
<td>-0.146</td>
<td>0.644*</td>
<td>0.565*</td>
<td>0.15</td>
<td>-0.123</td>
<td>0.538*</td>
<td></td>
</tr>
<tr>
<td>ABTSf</td>
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<td>-0.083</td>
<td>0.047</td>
<td>-0.404</td>
<td>0.051</td>
<td>0.374</td>
<td>-0.092</td>
<td>-0.113</td>
<td>0.141</td>
<td>0.49</td>
<td></td>
</tr>
<tr>
<td>DPPHf</td>
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<td>0.171</td>
<td>-0.078</td>
<td>-0.134</td>
<td>-0.441</td>
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<td>0.536*</td>
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<td></td>
</tr>
<tr>
<td>FRAPf</td>
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<td>0.595*</td>
<td>0.077</td>
<td>-0.32</td>
<td>-0.208</td>
<td>0.071</td>
<td>0.638*</td>
<td>-0.226</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>HSRAf</td>
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<td>-0.131</td>
<td>-0.452</td>
<td>-0.18</td>
<td>0.224</td>
<td>0.312</td>
<td>-0.247</td>
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<td></td>
<td></td>
<td></td>
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*Significant at $p < 0.05$.

Figure 3: Principal component analysis (PCA) shows the relationship free, bound phenolics, and their antioxidant activities. Correlation (a) between variables and factors and biplot (b).
and FRAPf ($r = 0.595$) and between DPPHF and FRAPb ($r = 0.536$) assays. In addition, a significant positive correlation was found between ABTSb and HRSAb ($p < 0.05$) as can be seen in Table 6. The results were in agreement with Zhang et al. [50], who found a significant relationship between ABTS, DPPH, and phenolics both bound and free forms of phenolic contents. However, there was no significant correlation between DPPH, FRAP, and flavonoid contents both free and bound forms of polyphenols. These results were in contrast with those obtained by Liyana-Pathiraka and Shahidi [39]. Free phenolic compounds showed a positive relationship ($p < 0.05$) between Tanf and HRSAb ($r = 0.538$) and ABTSb ($r = 0.565$). However, Flab showed only a significant positive correlation with Tanf ($r = 0.644$). The positive correlation of polyphenolics and their antioxidant activity was reported by Savlak et al. [51].

### 3.6. Principal Component Analysis (PCA)

PCA showed clearly the separation between bound and free phenolic content and their antioxidant activities (Figure 3). Figure 3(a) shows that the phenolic compounds and antioxidant activities quantified in the gari samples were reduced into two main components (PC1 and PC2). Free phenolics showed the opposite side of bound phenolic antioxidant activities except FRAPb (Figure 3(b)). F1 and F2 explain 46.21% of total data variance, with PC1 alone accounting for 27.69% of the observed variations. The variables which mainly contributed positively (F loading $> 0.62$) (Table 7) to PC1 are bound polyphenols forming antioxidants conferred by FRAP. However, PC2 accounts for 18.52% of the observed variations for free flavonoids; ABTS and DPPH contribute positively (F loading $> 0.77$) (Table 7). Thus, gari which has contributed mostly to the antioxidant capacities with their phenolics (bound and free) is divided into two groups according to F1 and F2 components (Figure 3(b)).

### 4. Conclusions

The present study revealed for the first time new knowledge on gari samples of bound and free phenolic compounds and their antioxidant activities. This study specifically demonstrated that gari samples were rich in phenolic contents and both free and bound forms showed antioxidant activity. However, the bound forms of phenolic compounds had a higher antioxidant activities than the free form. Thus, consumers of cassava gari can get health benefits to improve their well-being. It is recommended to harvest and process gari in dry season than in rainy season in order to maximize the antioxidant quality.

### Data Availability

Data are available on request (LA).

### Conflicts of Interest

The author declares no conflict of interest.

### Authors’ Contributions

LA conceived the experiments and designed the experiments, performed the experiments, analyzed and interpreted the data, and wrote the paper.

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