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PHYSICOCHEMICAL PROPERTIES OF GARLIC (*ALLIUM SATIVUM*) PASTE TREATED WITH ASCORBIC AND CITRIC ACIDS

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Abstract: The prime objective of the study was to develop a preservation method for garlic paste that could prevent adverse quality changes and render the paste more shelf stable. Three separate batches of fresh garlic bulbs of two Sudanese varieties (Dongla and Berber) harvested in December 2011were collected, peeled manually, separated into individual sound cloves, divided into 5 equal portions and crushed in a blender until a smooth puree was obtained. Before crushing, portions were assigned randomly to chemical treatments (T_0 - T_4). Each garlic treated portion was subdivided into 2 equal portions, packed in glass containers,, stored at 25°C or 40°C for 6 months and analyzed at an interval of 2 months. Parameters measured were, proximate composition, pH, viscosity, sugars, total soluble solids (TSS), tannic acid and carbohydrates (CHO). Berber *var*. had high ($p<0.05$) tannins but numerically low pH than Dongola *var*. These two components could render Berber *var*. less vulnerable to spoilage than Dongola. Irrespective of storage period, Dongola *var*. had higher ($p<0.05$) reducing and total sugars while Berber had higher ($p<0.05$) non reducing sugar. Also Irrespective of the chemical treatments (T_0 - T_4) moisture, fat, ash and crude fiber contents increased ($p<0.05$) while TSS, CHO and protein contents decreased ($p<0.05$) with the increase of the storage period. Treatments T_0 - T_4 caused variable effects increasing (TSS and crude fiber) or decreasing (moisture) some of the parameters measured.
Keywords: Garlic paste, ascorbic, citric, acids, physicochemical, properties.

INTRODUCTION

Garlic is an important cash crop in Sudan particularly in the Northern states (Dongla and Berber) and used as a seasoning agent in many foods around the country. The total annual production of garlic of Sudan is about 16,000 MT. In spite of the fact that garlic is among the major crops having a good prospect of earning foreign currency and of high nutritive and medicinal values, only few studies on its preservation and processing potentialities had been carried out. Cardiovascular disease is increasing rapidly in Sudan and it is now a public health concern. It has a major socio-economic impact on individuals, families and societies in terms of healthcare costs, work absenteeism and national productivity (Opie and Mayosi, 2005). Also the International Agency for research on cancer reported that the incidence rates of oral cancer in Sudan are 3.7% for men and 2.6% for women (IARC, 2008). Garlic and

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garlic extracts are believed to possess beneficial effects for the prevention of cardiovascular diseases (Koscielny, *et al.*, 1999; Rahman, 2001; Steiner and Li, 2001). Studies have shown that garlic also provides protection to the cardiovascular system by inhibiting platelet aggregation, protecting blood vessels and lipoproteins from damaging effects of free radical oxidation, and reducing serum cholesterol levels by inhibiting cholesterol synthesis (Piscitelli, *et al.,* 2002; Agarwal, 1996). Cancer preventative properties of garlic have also been reported (Ejaz, *et al.*, 2003). Epidemiologic studies have revealed the lower risk of stomach cancer in people with high garlic intake (Galeone *et al.*, 2006). Main pharmacological effects of garlic are attributed to its organosulphur compounds (Tapiero, *et al.* 2004).

The garlic paste manufacturing process dates back 40 years and stems from the need to find a product with an industrial or semi-industrial process able to compete in the international market. Another advantage is that the surplus from the nonmarketable part of the harvest (broken-up bulbs, loose cloves, etc.) may be used for paste manufacture. The consumption of processed garlic products (e.g. chopped and fried) has considerably increased over the last few years, probably due to its ease of use in comparison to fresh garlic (Oliveira *et al.*, 2004).

The main objective of the present work was to study the effect of inclusion of ascorbic and citric acids or their blend in the processing of GP stored for 6 months on its physicochemical characteristics.

MATERIAL AND METHODS

Preparation of garlic paste

Three batches of each fresh garlic bulbs of two Sudanese cultivars (Dongla and Berber) were harvested in December 2011, collected, packed and transferred to the Department of Food Science and Technology, Faculty of Agriculture, University of Khartoum, Sudan. The garlic bulbs were peeled manually, separated into the individual sound cloves, cut in half and inner green shoots removed. Prepared garlic cloves were divided into 5 equal portions and crushed in a blender until a smooth puree was obtained in about 3 min. Before crushing, portions were assigned randomly to one of the following 5 chemical (organic acids) treatments:T_0 = Control (no chemical added) , T_1= Ascorbic acid 0.5mg/g , T_2 = Citric acid 2mg/g , T_3 = Ascorbic acid 0.25 mg/g + Citric acid 1mg/g and T_4 = Ascorbic acid 0.5mg/g + Citric acid 2mg/g. The chemicals or their blends were added during crushing. The garlic paste was packed in aseptic glass containers and tightly closed. Samples were stored at $25^{O}C$ and $40^{O}C$ for six months. Sample for analysis was taken at an interval of 2 months.

Proximate analysis:

Moisture, crude protein, fat, ash and crude fiber contents of garlic paste were determined according to the AOAC (2003).

Physicochemical properties

pH-value

The pH of garlic was determined in 10% solution of sample using a pH meter (Model L. Pusl muchen 15-1260, Germany).

Total soluble solids (TSS)

Total soluble solids of garlic were determined according to AOAC (2003) using hand refractometer, and were expressed as ^{o}Brix.

Viscosity

The viscosity was determined according to the method of Gidley, *et al*. (1991).

Sugar contents:

The reducing and non-reducing sugar contents of garlic samples were determined according to AOAC (2003). Total sugar was calculated by summation of reducing and non-reducing sugar contents.

Determination of tannic acid

Tannin content (TC) of garlic samples were estimated using modified vanillin-HCl in methanol as described by Price *et al.* (1978). About 0.2g of garlic paste was placed in 100ml conical flask. 10ml of 1%Hcl in methanol (v/v) were added, the contents were mechanically shaken for 20min and centrifuged at 2500rpm for 5min. One ml of supernatant was pipetted into a test tube and 5ml of vanillin-Hcl reagent (mixing equal volume of concentrated 8% Hcl in methanol and 1% vanillin in methanol) were added. The optical density was read at 500nm using a colorimeter (Lab system Analyzer 9 filters, Mitra and Bros. Pvt. Ltd) after incubation at $30^{o}C$ for 20 min. A blank sample was carried out with each run of samples. A standard curve was prepared expressing the result of tannic acid, i.e. amount of tannic (mg / ml) which gives color intensity equivalent to that given by tannin after correcting for blank.Tannin content(TC) was calculated from the following equation:

$$\text{TC}(\%) = \frac{C \times 10 \times 100}{200}$$

Where:

C = concentration corresponding to optical density.

10 = volume of extract in ml; 200 = sample weight in mg.

RESULTS AND DISCUSSION

Effect of variety and temperature on the physicochemical properties

The effect of variety and temperature on the pH, viscosity and tannic acid of garlic paste (GP) are shown on Table 1. Irrespective of temperature, variety had no effect ($p>0.05$) on the pH of GP as the two varieties (Dongola and Berber) had similar pH values, however it significantly affected both viscosity and tannic acid where Berber had the highest ($p<0.05$) values than Dongola. Except for tannic acid, GP stored at 40^{O}C had the highest ($p <0.05$) pH-value (4.16) and viscosity (0.056) compared to those stored at 25^{O}C, while the latter resulted in the highest ($p<0.05$) tannic acid content (1.76%). The tannic acid value found in this study was way higher than that reported earlier by Nwinuka *et al.*(2005). The pH values of the current study were quite low compared to that (5.80) reported by USFDA (2007). The observed discrepancies between the two studies could be attributed to the fact that, in the current study GP was mixed with organic acids (ascorbic acid, citric acid or their blends). Addition of 2.1g citric acid/ 100g garlic lowered the initial pH to 4.0 (Berbari *et al.,* 2003). In addition, varietal differences, processing and storage condition could have accounted partially to such discrepancies. Among other factors green pigmentation of GP is influenced by pH. Since the optimum pH for garlic greening is approximately 5.5, addition of organic acids to the GP could serve as a barrier against green pigmentation formation. It is interesting to note that at any one temperature studied, Berber *var.* had always higher viscosity than Dongola *var*. Viscosity is central to texture and mouth feel of many food types including garlic. As such, consumers could sensorially perceive Berber *var.* better than Dongola *var*.

The effects of variety and temperature on reducing, non-reducing and total sugars of GP are shown on Table 2. Irrespective of temperature, Dongola *var.* had higher ($p<0.05$) reducing (35.13%) and total (44.79%) sugar contents than Berber *var.* (30.99% and 41.62%, respectively). On the other hand Berber had higher ($p<0.05$) non-reducing sugar content (10.12%) than Dongola (9.70%). Both varieties had non-reducing sugar content that fall within the range 7.5 – 13.9 g/100g reported for onion by DOGR (2010). Garlic paste stored at 40^{O}C had always the highest ($p<0.05$) reducing, non- reducing and total sugars than the ones stored at 25^{O}C. On the average Sudanese garlic had higher total sugar content than the

Romanian one. Berar (1998) found that the Romanian garlic contain 20 – 26% total sugar. Apparently this wide range is due to the genotypic variation of the tested samples.

Effect of chemical treatments and storage periods on chemical composition

The effect of chemical treatments (T_0-T_4) and storage periods (2- 6 months) on the moisture, protein and fat contents are shown on Table 3. Irrespective of the treatments (T_0-T_4) moisture and protein contents of GP increased and decreased respectively with the increase of storage period. Garlic paste stored for 6 months had significantly ($p<0.05$) the highest moisture (64.88%) and the lowest protein (2.19%) contents compared to that stored for 2months (60.61% moisture and 4.60 % protein). After the 4^{th} month of storage, the fat content of GP increased with the increase of storage period. Also irrespective of the storage period, the chemical treatments (T_0-T_4) affected the moisture content of GP significantly where untreated GP (T_0) had the highest ($p< 0.05$) moisture content (63.88%) compared to the treated ones (T_1-T_4). The values obtained in this study were within the range of values obtained by Benkeblia (2007), who reported that garlic contain 60-70% moisture. The moisture content of T_0 was comparable to the values (66.57%) reported by Odebunmi *et al*. (2010) for the Nigerian garlic and to that (65%) reported by Blumenthal and Mark (2000). Also Topno *et al.* (2011) found that GP contained 63.04% moisture.

The protein and fat contents were significantly ($p<0.05$) affected by the treatments to which GP was subjected. Garlic paste treated with T_2 had the highest fat content (1.47%) compared to that treated with T_4 (1.20%). The value obtained in this study for the control sample (1.43%) was higher than that obtained by Blumenthal and Mark, (2000) or that of Nwinuka *et al.* (2005) who reported that garlic contain 0.15% and 0.68% fat content respectively. Generally the chemical treatments tended to decrease the protein content of GP. The different treatments (T_0-T_4) had protein contents in the range of 3.17% - 3.84% which was lower than the value obtained by Banerjee *et al*. (2003), who reported that garlic contain 5% protein content. Irrespective of storage period, control garlic paste had a protein content of 3.42% almost 1.7 times higher than that proposed by HAS (2006) guide. Also it is less than that reported for the Nigerian garlic (17.35% crude protein) according to Nwinuka *et al.,* (2005) or that (17.5% crude protein) reported in the Encyclopedia of Chemical Technology (1980). Spanish garlic had a protein content of 18.83% (Cardelle- Cobas *et al*., 2005). Discrepancies in the findings of the different studies quoted here could be a result of some investigators had expressed their protein content on dry weight basis, while that of Banerjee *et al*. (2003) and the current study were on wet weight basis. Also varietal differences, geographical locations and agronomical practices could account partially to the observed differences.

The interactive effect of treatment and storage on the crude fiber, ash and TSS are shown on Table 4. Results indicated that storage period had a significant ($p<0.05$) effect on the amount of crude fiber. Garlic paste stored for 6 months had significantly ($p<0.05$) the highest (4.86%) crude fiber compared to that stored for 2months (3.52%). Similarly the crude fiber of GP was significantly ($p<0.05$) affected by the chemical treatment to which GP was subjected. Garlic paste treated with T_4 had significantly ($P<0.05$) the highest (4.37%) crude fiber compared to that of T_0 (3.96%). USDA (2004), Odebunmi *et al.* (2009) and Blumenthal and Mark (2000) reported that garlic contain 2.10%, 0.73 % and 1.5 % crude fiber respectively. While the ash content of GP increased with the increase of the storage period, the TSS showed an opposite trend as it decreased with the increase of the storage period particularly after the 4^{th} month of the storage. Chemical treatments (T_0 -T_4) had no clear effect on the ash content of GP. However within treatments (T_0 -T_4) ash contents increased with the increase of the storage period while TSS decreased with the increase of the storage period. The value obtained in this study was similar to the value obtained by Casado *et al*. (2004), who reported that, the ash content of garlic ranged between 2.65% and 8.40%.

Irrespective of the treatments (T_0-T_4) carbohydrate content of GP decreased with the increase of storage period (Table 5). Garlic paste stored for 6 months had significantly ($p<0.05$) the lowest carbohydrate (20.94%) content compared to that stored for 2months (26.80%). The chemical treatments (T_0-T_4) had variable effects on the carbohydrate content of GP. Treatments T_2, T_3 and T_4 registered the highest ($p< 0.05$) carbohydrate contents (24.65%, 24.36% and 25.64% respectively), while T_0 and T_1 showed the lowest values (23.11% and 23.41% respectively). The value obtained in this study was higher than the value obtained by Sowbhagya *et al*. (2009) who reported that garlic contain 21% carbohydrates but lower than that (28%) stated by HSA (2006).

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Table 1. Effect of garlic variety and temperature on pH-value, viscosity and tannic acid composition of garlic paste.

Temperature (°C)	Variety *		Average temp. ***
	Dongola	Berber	
pH-value			
25°C *	3.92±0.22 ^{b}	3.90±0.25 ^{b}	3.91 ^{B}
40°C	4.18±0.60 ^{a}	4.14±0.51 ^{a}	4.16 ^{A}
Average variety **	4.05 ^{A}	4.02 ^{A}	
Viscosity (cp)			
25°C	0.05291±0.03 ^{b}	0.05251±0.03 ^{c}	0.053 ^{B}
40°C	0.05120±0.03 ^{d}	0.06171±0.04 ^{a}	0.056 ^{A}
Average variety	0.052 ^{B}	0.057 ^{A}	
Tannic acid (%)			
25°C	1.77±0.73 ^{b}	1.75±0.74 ^{b}	1.76 ^{A}
40°C	1.70±0.71 ^{c}	1.82±0.76 ^{a}	1.74 ^{B}
Average variety	1.72 ^{B}	1.79 ^{A}	

*Means in the same columns and rows bearing same superscript small letters are not significantly different (P>0.05)

**Means in the same row with the same superscript capital letters are not significantly different (P>0.05).

***Means in the same column bearing different superscript capital letters are significantly different (P<0.01).

Table 2. Effect of garlic variety and temperature on reducing sugars (%), non-reducing sugars and total sugars composition of garlic paste

Temperature (°C)	Variety *		Average temp. ***
	Dongola	Berber	
reducing sugars (%)			
25°C *	31.27±4.93 ^{c}	27.51±5.52 ^{d}	29.39 ^{B}
40°C	38.99±14.08 ^{a}	34.46±9.24 ^{b}	36.73 ^{A}
Average variety **	35.13 ^{A}	30.99 ^{B}	
Non-reducing sugars (%)			
25°C	8.41±4.19 ^{d}	10.30±4.54 ^{b}	9.29 ^{B}
40°C	11.16±0.21 ^{a}	9.94±5.55 ^{c}	10.53 ^{A}
Average variety	9.70 ^{B}	10.12 ^{A}	
Total sugars (%)			
25°C	39.68±5.09 ^{c}	38.30±4.19 ^{d}	38.57 ^{B}
40°C	50.14±11.08 ^{a}	44.93±9.06 ^{b}	47.84 ^{A}
Average variety	44.79 ^{A}	41.62 ^{B}	

*Means in the same columns and rows bearing same superscript small letters are not significantly different (P >0.05)

**Means in the same row with the same superscript capital letters are not significantly different (P>0.05).

***Means in the same column with different capital letters differ significantly (P<0.01).

Table 3. Effect of chemical treatments and storage periods on moisture, protein and fat contents of garlic paste

Treatments	Storage period (months) *			Average treat. ***
	2	4	6	
Moisture content (%)				
T_0 *	62.26±1.29^{g}	63.60±1.27^{e}	65.78±0.74^{a}	63.88^{A}
T_1	61.29±1.29^{jh}	63.21±1.32^{f}	65.38±1.00^{b}	63.29^{B}
T_2	59.88±1.25^{j}	64.06±1.20^{d}	64.29±0.83^{d}	62.74^{CD}
T_3	60.40±1.37^{i}	63.56±1.67^{e}	64.85±0.85^{c}	62.94^{C}
T_4	59.21±1.11^{k}	62.10±1.45^{g}	64.11±0.85^{d}	61.81^{D}
Average storage **	60.61^{C}	63.31^{B}	64.88^{A}	
Protein content (%)				
T_0	4.15±1.06^{bc}	3.87±1.05^{d}	2.23±0.32^{g}	3.42^{AB}
T_1	5.23±0.24^{a}	3.81±0.81^{d}	2.46±0.14^{f}	3.84^{A}
T_2	4.59±0.66^{b}	3.21±0.83^{e}	2.11±0.18^{g}	3.30^{B}
T_3	4.29±0.80^{c}	3.18±1.13^{e}	2.05±0.23^{g}	3.17^{BC}
T_4	4.72±0.59^{b}	3.05±0.96^{e}	2.10±0.33^{g}	3.29^{B}
Average storage	4.60^{A}	3.42^{B}	2.19^{C}	
Fat content (%)				
T_0	1.61±0.36^{cde}	1.39±0.16^{g}	2.29±0.14^{a}	1.43^{A}
T_1	1.31±0.23^{f}	1.57±0.36^{g}	2.03±0.45^{b}	1.30^{AB}
T_2	1.53±0.31^{cdefg}	1.45±0.26^{g}	2.42±1.02^{a}	1.47^{A}
T_3	1.49±0.36^{defg}	1.53±0.18^{g}	1.78±0.28^{g}	1.27^{AB}
T_4	1.38±0.29^{ef}	1.57±0.30^{g}	1.65±0.29^{cd}	1.20^{B}
Average storage	1.46^{B}	1.50^{B}	2.03^{A}	

*Means in the same columns and rows bearing same superscript small letters are not significantly different (P>0.05)

**Means in the same row with the same superscript capital letters are not significantly different (P>0.05).

***Means in the same column bearing different superscript capital letters are significantly different (P<0.01).

T_0 = control;T_1 = Ascorbic acid (0.5mg/g);T_2 = Citric acid (2mg/g);T_3 = Ascorbic acid (0.25mg/g) + Citric acid (1mg/g);T_4 = Ascorbic acid (0.5mg/g) + Citric acid (2mg/g)

Table 4: Effect of chemical treatments and storage periods on crude fiber, ash content and total soluble solids composition of garlic paste

Treatments	Storage period (months) *			Average treat. ***
	2	4	6	
Crude fiber (%)				
T_0 *	3.72±0.68^{g}	3.43±0.25^{h}	4.72±0.46^{b}	3.96^{B}
T_1	4.06±0.62^{ef}	4.24±0.91^{de}	4.53±0.18^{c}	4.28^{A}
T_2	3.07±0.31^{i}	3.91±0.99^{f}	4.95±0.78^{a}	3.98^{B}
T_3	3.06±1.32^{i}	4.47±0.95^{c}	5.07±0.60^{a}	4.20^{AB}
T_4	3.68±0.42^{g}	4.42±0.95^{cd}	5.01±0.72^{a}	4.37^{A}

Average storage**	3.52^{C}	4.09^{B}	4.86^{A}	
Ash content (%)				
T_0	2.93±0.14^{g}	3.77±0.48^{e}	5.30±0.42^{ab}	4.00^{A}
T_1	2.83±0.37^{g}	3.94±1.12^{d}	5.22±0.42^{b}	3.89^{AB}
T_2	2.94±0.17^{g}	3.66±0.19^{f}	4.99±0.24^{c}	3.86^{AB}
T_3	2.86±0.41^{g}	4.00±0.39^{d}	5.33±0.29^{a}	4.06^{A}
T_4	2.91±0.33^{g}	3.64±0.16^{f}	5.08±0.15^{bc}	3.88^{AB}
Average storage	2.89^{C}	3.80^{B}	5.18^{A}	
Total soluble solids (%)				
T_0	43.01±0.75^{d}	42.83±0.79^{e}	40.06±0.76^{j}	41.97^{D}
T_1	43.51±0.96^{b}	43.53±1.34^{b}	40.67±1.45^{i}	43.90^{A}
T_2	43.63±0.51^{b}	43.58±2.41^{b}	41.21±0.66^{h}	42.81^{BC}
T_3	43.08±1.82^{d}	42.70±0.52^{f}	41.66±0.58^{g}	42.40^{C}
T_4	43.24±1.42^{c}	43.84±0.91^{a}	41.76±1.51^{g}	42.95^{B}
Average storage	43.29^{A}	43.25^{A}	40.95^{B}	

*Means in the same columns and rows bearing same superscript small letters are not significantly different (P>0.05)

**Means in the same row with the same superscript capital letters are not significantly different (P>0.05).

***Means in the same column with different capital letters differ significantly (P<0.01).

T_0 = control. T_1 = Ascorbic acid (0.5mg/g); T_2 = Citric acid (2mg/g);T_3 = Ascorbic acid (0.25mg/g) +Citric acid (1mg/g); T_4= Ascorbic acid (0.5mg/g) + Citric acid (2mg/g)

Table 5: Effect of chemical treatments and storage periods on carbohydrates of garlic paste

Treatments	Storage period (months)*			Average treat.***
	2	4	6	
Carbohydrates (%)				
T_0^{*}	24.72±1.28^{e}	24.94±1.25^{d}	20.17±1.03^{k}	23.11^{C}
T_1	25.27±1.19^{c}	24.57±2.71^{e}	20.38±1.14^{j}	23.41^{C}
T_2	28.00±0.50^{a}	24.70±1.80^{e}	21.25±1.56^{h}	24.65^{B}
T_3	27.90±2.30^{a}	24.26±2.23^{f}	20.92±1.33^{i}	24.36^{B}
T_4	28.10±1.35^{a}	26.23±2.11^{b}	21.98±1.27^{g}	25.64^{A}
Average storage**	26.80^{A}	24.94^{B}	20.94^{C}	

*Means in the same columns and rows bearing same superscript small letters are not significantly different (P>0.05)

**Means in the same row with the same capital letters are not significantly different (P>0.05).

***Means in the same column with different capital letters differ significantly (P<0.01).

T_0 = control; T_1 = Ascorbic acid (0.5mg/g); T_2 = Citric acid (2mg/g); T_3 = Ascorbic acid (0.25mg/g) + Citric acid (1mg/g); T_4= Ascorbic acid (0.5mg/g) + Citric acid (2mg/g)