## Effect of Lime Soaking and Cooking (Nixtamalization) on the Proximate, Functional and Some Anti-nutritional Properties of Millet Flour

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#### Abstract

The effect of lime soaking and cooking on the functional, proximate, and antinutritional properties of millet flour was studied. Flour samples were produced from soaked millet grains and cooked millet grains. Portions of millet grains were soaked in water and in 1% lime solution for 24h and some were cooked for 30minutess in both water and in 1% lime solution. At the end of soaking and cooking, the grains were dried; milled; sieved with a 0.25 mesh screen; and packaged in white high density polyethylene bags. Cooking millet grains in lime solution prior to processing into flour resulted in a flour that contains significantly (p < 0.05) higher protein; ash; crude fibre; water absorption capacity; pH; emulsion capacity and stability; hygroscopicity; and swelling power; and significantly (p < 0.05) lower fat; oil absorption capacity; tannin; trypsin inhibitor and hydrogen cyanide than flours obtained from untreated millet grains and millet grains cooked in water. Furthermore, flour obtained from millet grains soaked in lime solution contains significantly (p<0.05) higher water absorption capacity; hygroscopicity; and swelling power; and significantly lower protein; phtyic acid; tannin; and trypsin inhibitor than flours obtained from untreated millet grains and millet grains soaked in water.

*Keywords*: Anti-nutritional properties, functional properties, soaking, nixtamalization.

#### Introduction

Nixtamalization refers to a process of preparing maize (corn) in which the grain is cooked and soaked in alkaline solution, usually lime solution, and then dehulled. The term 'nixtamalization' also refers to the removal of the pericarp from any grain using an alkaline process. The basic process begins by cooking whole grains in water with lime and steeping the cooked grains for 12-16h in large tanks. The steeped grain is called 'nixtamal' and the cooked steep liquid, rich in maize solids, is called 'nejayote' (Sahai, Surjewan,Mua, Buendia, Rowe and Jackson, 2000). The process of nixtamalization is popular in Mexico and Central America and

ess of nixtamalization is and Central America and has been applied to corn for centuries (Bressani, Benavides, Aceredo and Ortiz, 1990). Grains subjected to nixtamalization process have several benefits over unprocessed grains for food preparation: they are more easily ground; their nutritional value is increased; flavour and aroma are improved and mycotoxins (Sefa-Dedeh, are reduced Cornelius, Sakyi and Afoakwa, 2004). These benefits make nixtamalization a crucial preliminary step for further processing of grains into food products. Unleavened bread (tortilla) is a common food in central and South America produced from sorghum cooked in lime solution (Asiedu 1989). In nixtamalization, dried cereal grains are cooked in an alkaline solution at or near its boiling point. After cooking, the grains are steeped in

the cook liquor for a length of time. Cooking and soaking varies according to local tradition and type of food to be prepared. During cooking a number of chemical changes take place in the grains: the kernels soften and their pericarp loosen causing the plant cell wall including hemicelluloses and pectin to become soluble; the grain is hydrated and absorbs the alkali used from the cooking solution; starches swell and gelatinize and some disperse into the liquid. After steeping, the alkaline liquid (nejayote) containing dissolved components of the maize, is discarded. The kernels are washed thoroughly of the remaining *nejayote* which has an unpleasant flavour (Watcher, 2003). The kernels are then dehulled to remove the pericarp and the germ. The grain is called *nixtamal* and it has many uses contemporarily and historically. Whole nixtamal may be used fresh or dried for later use.

Millet is an indispensable food for millions of people inhabiting the semi arid tropics. It is used primarily for human food and remains a major source of calories and a vital component of food security in the semiarid areas in the developing world (FAO, 1996). It is a short season crop and an efficient water user. It is therefore grown in many areas of the world which have limited rainfall (Bishop, Carter, Chapman and Bennett, 1982). In West African semi-arid tropics, cereals occupy nearly seventy percent of total cultivated area and engage fifty to eighty percent of total farm level resources (Matlon, 1990). In a comparison between rice, millet, sorghum and maize on the basis of utilization. Reardon (1993) reported that the combined share of millet and sorghum exceeds that of maize in the urban diets of the Sahel and is second to rice only because of the relatively low cost of imported rice and ease of cooking and processing into fast food. The worker further stated that there is the need to develop suitable processing and milling methods for sorghum and millet in order to produce innovative ready-to-eat products from these grains that could be sold by street vendors and markets would open up new avenues of utilization that would reduce the dependence on foreign rice.

Due to the high dependence on millet and other cereals (particularly maize and sorghum) in developing countries (particularly tropical Africa) and coupled with their low nutritive value. there is continuous investigation into methods of improving the nutritional value of diets derived from them. This research work is aimed at investigating the effect of soaking and cooking millet in solution (nixtamalization) lime on the proximate. functional and anti-nutritional factors of millet flour with a view to improving food products derived from millet flour.

## Materials and Methods

Pearl millet grains were purchased from Minna central market, Minna, Niger State, Nigeria while calcium hydroxide (Ca (OH)<sub>2</sub>) and all other chemicals were obtained in the Food Science and Nutrition laboratory, Federal University of Technology, Minna and the Root and Tuber Crops Research Institute, Umudike, Abia State, Nigeria.

### **Preparation of Samples**

Millet grains were manually cleaned to remove husk, stems, damaged and discoloured seeds. This was achieved by winnowing, hand picking and washing with tap water after which they were dried in a hot air oven at a temperature of  $60^{\circ}$ C for 90 mins.

# Preparation of Untreated Millet Flour (Control)

400g of millet grains were dry milled, using a disc attrition mill and then sieved using a 0.25mm mesh screen. The flour was then packaged in a white high density polyethylene bag and stored in the refrigerator at a temperature of  $4^{\circ}$ C.

#### Preparation of Flour from Soaked Millet Grains

400g of millet grains were soaked in 2,000ml of 1% lime solution while another 400g was soaked in tap water for 24h after

which the grains were thoroughly washed with tap water and drained. The grains were then dried in a hot air oven at a temperature of  $60^{\circ}$ C for 90 mins. The grains were dry milled using a disc attrition mill after which they were sieved using a 0.25mm mesh screen. Both flours were then packaged using a white high density polyethylene bag and stored in a refrigerator at a temperature of  $4^{\circ}$ C.

#### **Preparation of Flour from Cooked Millet Grains**

400g of millet grains were boiled/cooked in 2,000ml of 1% lime solution while another 400g was boiled/cooked in tap water for 30 mins after which they were steeped in plastic containers for 24h. After steeping, the grains were thoroughly washed to remove the pericarp. They were then dried in a hot air oven at  $60^{\circ}$ C for 90 mins and then dry milled using a disc attrition mill. Both flours were sieved using a 0.25mm mesh screen and then packaged in white high density polyethylene bag and stored in a refrigerator at a temperature of  $4^{\circ}$ C.

#### Analysis of Functional Properties. Loose and Packed Bulk Densities

The loose and packed bulk densities of the flours were determined using the methods described by Akpapunam and Markakis (1981). 50g portions of the samples were weighed into a 100ml measuring cylinder and the volume noted. The loose bulk density was then calculated, thus:

Loose bulk density = mass of sample / volume of sample.

For packed bulk density 50g of each of the samples was weighed into a 100ml measuring cylinder. The cylinder was tapped 40 times and the packed bulk density was calculated as weight per unit volume: Packed bulk density = mass of sample / volume of sample after tapping.

#### **Oil and Water Absorption Capacities**

The oil and water absorption capacities were determined using the methods described

by Okezie and Bello (1988). 1g of each sample was mixed with 20ml distilled water (oil in the case of oil absorption capacity) in a flask shaker and then centrifuged at 2,000 rpm for 1h. Water/oil absorbed by samples was calculated as the difference between the initial and final volumes of water/oil.

#### Hygroscopicity

The hygroscopicity of flour samples was determined as described by Bhatty (1988). About 5g of each flour sample was exposed to ambient conditions  $(32\pm2^{0}C \text{ and } 70\text{-}75\% \text{ relative humidity})$ . Hygroscopicity was expressed as the percentage of weight gained by samples after 48h.

#### **Swelling Power**

Swelling power of flour samples was determined using the procedures described by and Ooraikul and Moledina (1981). 10% slurry of each sample was prepared using distilled water. The slurries were agitated in test tubes for 30 mins and then centrifuged (NAAFCO centrifuge, CFC-300 England) for 15 mins at 1,000 rpm after which they were decanted and 10ml distilled water was added to the tubes. Swelling power (SP) was calculated, thus: SP = (total volume – 10) x 4 (ml/g).

#### **Emulsion Capacity and Stability**

The emulsion capacity and stability of the flours were determined using the method of Yatsumatsu, Sawada, Moritaka, Misaka, Toda and Woda (1972). About 2g of each of the samples was weighed and 15ml distilled water and 10ml vegetable oil (Gino vegetable oil) was added to each sample in a centrifuge tube. These were then centrifuged at 1,600 rpm for 10 mins and the height of the emulsified layer was measured. Emulsion capacity was calculated as the ratio of the height of the emulsified layer to the height of the whole layer (material). Emulsion stability was calculated as the ratio of the height of the emulsified layer to the total height of the material after heating at 80°C for 30 mins; cooling under running water for 15 mins; and centrifuging at 1,600 rpm for 15 mins.

#### Analysis of Proximate Composition

The moisture, ether extract, crude protein, crude fibre, ash, carbohydrate and pH values of the flours were all determined using the method of AOAC (2000).

#### Analysis of Anti-nutritional Factors

The trypsin inhibitor and cyanogenic glucoside (hydrogen cyanide) contents of the flours were determined using the method described by AOAC (2000) while the tannin and phytate contents were determined using the method of Pearson (1976).

#### **Statistical Analysis**

All determinations were carried out in triplicates and the means were subjected to one-way analysis of variance by means of MINITAB 14 statistical software.

### **Results and Discussion**

Table 1 shows the proximate composition of millet flours. Dry matter values ranged from 89.51 to 93.75 with flour from millet cooked in lime having the lowest value and flour from untreated millet having the highest value. Differences in dry matter were not significant (p>0.05).

Crude protein values ranged from 11.57% to 14.57%. Cooking of millet grains in lime solution resulted in a significant (p < 0.05) increase in the protein content of the flour. This is in agreement with Bressani and Scrimshaw (1958) who reported that protein content of maize increased from 9.6% to 10.3% when maize was nixtamalized. This was attributed to a concentration effect. Gomez, Rooney, Waniska and Plugfelder (1987) and Serna-Saldivar, Knabe, Rooney and Tanksley, (1987) also reported an increase in the amount of protein when alkaline cooked corn products were compared to original grain. This indicates that in millet-consuming areas where protein deficiency is prevalent, lime cooking of millet will be an advantage.

Fat values ranged from 4.02% to 7.43% with flour from millet cooked in lime having the lowest value and flour from millet soaked in water having the highest value. Lime cooking significantly (p < 0.05) decreased the fat content of flour. This may be because elevated temperatures and metallic ions (in this case Ca<sup>2+</sup>) facilitate fats oxidation and degradation (Charley and Weaver, 1998). This implies that the flour from millet cooked in lime will be the least susceptible to rancidity under similar storage conditions and consequently possess better flavour and aroma after storage.

Ash values ranged from 1.12% to 3.23% with flour from untreated millet having the least value and flour from millet cooked in lime having the highest value. Lime cooking significantly (p<0.05) increased the ash content of flour. This increase may be due to absorption of calcium ion (Ca<sup>2+</sup>) from the cooking medium. This means consumption of meals derived from lime-cooked millet is advantageous with respect to mineral content.

Crude fibre values ranged from 4.15% to 6.32% with flour from millet soaked in lime having the least value and flour from millet cooked in lime having the highest value. Cooking in lime solution significantly (p<0.05) increased the crude fibre of the flours. This may be due to interactions between ions (Ca<sup>2+</sup> and OH<sup>-</sup>) present in the cooking solution/medium and the constituents of the grains which could have produced some indigestible products thereby increasing the crude fibre content. This may be an advantage with respect bowel movement since crude fibre facilitates easy passage of waste from the bowel.

Carbohydrate value ranged from 71.95% to 77.13% with flour from millet cooked in lime having the least value and flour from millet soaked in lime having the highest value. Lime cooking significantly (p<0.05) decreased the carbohydrate content. The relatively low carbohydrate content of the lime cooked sample is probably due to its higher protein, ash and crude fibre contents since carbohydrate was determined by difference.

Table 2 shows the functional properties

of millet flours. The water absorption capacity values ranged from 3.10g/g to 5.41 g/g with flour from untreated millet having the lowest value and flour from millet cooked in water having the highest value. Apart from water soaking, other treatments significantly (p < 0.05) increased the water absorption values of the flours with flour from millet cooked in lime having the most significant increase. This may be due to its relatively higher and relatively lower protein and fat contents respectively. It could also be due to the facilitating effect of lime on gelatinization of its starch content. An advantage of high water absorption is that it facilitates easy digestion while a disadvantage is a relatively high water activity which will cause the food to be easily spoiled by micro-organisms.

Oil absorption capacity values ranged from 1.22 to 1.52g/cm<sup>3</sup> with flour from untreated millet having the least value and flour from millet soaked in water having the highest value. Though lime soaking significantly (p<0.05) increased the oil absorption capacity of flour, its value was still lower than those of the water treated samples.

Bulk density values ranged from 0.59g/cm<sup>3</sup> to 0.68g/cm<sup>3</sup> with flour from millet soaked in water having the highest value and flour from untreated millet having the lowest value. Loose bulk density values ranged from 0.50g/cm<sup>3</sup> to 0.53g/cm<sup>3</sup> with flours from untreated millet and millet soaked in water having the least values while flour from millet cooked in lime had the highest value. No significant (*p*<0.05) differences were observed in the bulk densities of the flours.

pH values ranged from 6.42 to 9.04 with flour from millet cooked in lime having the highest value. Lime soaking significantly (p<0.05) increased the pH of the flour. The pH of food products is an important parameter as it affects flavour and shelf life of products. The increase in the pH of the flour from millet cooked in lime solution is probably due to absorption and retention of lime. This implies that the flour may be more prone to spoilage under similar storage conditions due to its reduced acidity.

Emulsion capacity values ranged from 19% to 27% with flour from untreated millet

having the least value and flour from millet cooked in lime having the highest value. Emulsion stability values ranged from 20% to 26% with flour from untreated millet having the least value and flour from millet cooked in lime having the highest value. Lime cooking significantly (p < 0.05) increased both emulsion capacity and emulsion stability of flours. This could be due to its lower and higher fat and protein contents respectively. Nkonge and (1984);and Kato, Fivishige, Balance Matusdomi and Kobayashi (1985) reported that the emulsion capacity of a product is dependent on the oil content and protein concentration of the product. Furthermore, the efficiency of emulsion varies with concentration and solubility of protein (Achinewhu, 1983). The significantly higher Emulsion stability of lime-cooked millet suggests that its flour will much more easily be incorporated into food mixtures than flour from untreated millet.

Hygroscopicity values ranged from 30% to 38% with flour from millet soaked in water having the least value and flour from millet cooked in lime having the highest value. solution significantly Cooking in lime (p < 0.05) increased the hygroscopicity of the flours. This could be due to the presence of more hydrophilic compounds in this flour than in the other ones. It could also be due to its lower fat content. The ability of a food particle to absorb water on its surface, thus initiating reconstitution is an important parameter in the preparation of gruels. The high value observed indicates that flour from millet cooked in lime solution has this advantage. However, the ability to absorb water may make it more susceptible to spoilage coupled with its high pH (low acidity).

Swelling power values ranged from 2.17 to 4.50g/g with flour from untreated millet having the least value and flour from millet cooked in lime having the highest value. All treatments significantly (p<0.05) increased the swelling power of the flours but lime cooking had the most significant increase. This could be due to the reduced fat content of the flour: Zobel (1984) reported that fats may complex with starch and limit swelling; and its high water absorption capacity. A high swelling power will translate to more quantity of product from a given quantity of the flour.

Some of the anti-nutritional factors of millet flours are shown in Table 3. Phytic acid content ranged from 46.10mg/100g to 89.64 mg/100g with the least value in flour from millet cooked in water and the highest value in flour from untreated millet. All treatments significantly ( $p \le 0.05$ ) decreased the phytic acid contents. The reduction in phytic acid as a result of all treatments may be due to degradation of phytic acid by the enzyme phytase which is usually activated by soaking as well as the action of applied heat in the process of cooking. The result indicates that cooking in water is more effective in reducing the phytic acid content than cooking or soaking in lime solution.

Tannin contents ranged from 1.01g/Kg to 1.94g/Kg with flour from millet soaked in lime having the least value and flour from millet cooked in water having the highest value. All treatments significantly (p < 0.05)decreased the tannin content except water cooking of millet which significantly increased it. Price, Hagerman and Butler (1980) observed that tannin content of sorghum flour decreased when mixed into batter with a further reduction on cooking. Furthermore, Price and Butler (1977) reported that moisturizing grains with alkali several hours to processing or utilization was found to be quite effective in inactivating or detoxifying tannins in bird resistant sorghum.

Trypsin inhibitor values ranged from 5.31mg/g to 13.40mg/g with flour from millet cooked in lime having the least value and flour from millet cooked in water having the highest value. All lime treatments significantly (p < 0.05) decreased trypsin inhibitor contents of flours. This means the protein of flour from lime treated millet will be less inhibited (more biologically available) than that from water treated millet. Cooking, soaking and traditional methods of processing cause significant reduction in trypsin inhibitor activity (Akinyele, 1989; Egbe and Akinyele, 1990).

Cyanide values ranged from 7.80mg/100g to 16.05mg/100g with flour from millet soaked in water having the highest

value. Lime treatments significantly (p < 0.05) decreased the hydrogen cyanide content. The reduction in cyanide content of the flour samples may have been due to the effect of soaking and cooking which could have caused hydrogen cyanide to be lost during cooking and soaking.

#### Conclusion

Cooking millet grains in lime solution prior to processing into flour resulted in a flour that contains significantly higher protein, ash, crude fibre, water absorption capacity, pH, capacity emulsion and stability, hygroscopicity, and swelling power; and significantly lower fat, oil absorption capacity, tannin, trypsin inhibitor and hydrogen cyanide than flours obtained from untreated millet grains and millet grains cooked in water. Furthermore, flour obtained from millet grains soaked in lime solution contains significantly higher water absorption capacity: hygroscopicity; and swelling power; and significantly lower protein; phytate; tannin; and trypsin inhibitor than flours obtained from untreated millet grains and millet grains soaked in water. Consumption of meals derived from lime-treated millet flour as compared with untreated millet flour is advantageous with respect to nutrients. However, flour from untreated millet flour may keep better than flour from lime-treated millet.

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Parameters	UMF	WSMF	LSMF	WCMF	LCMF
Dry matter	93.75±2.27 <sup>a</sup>	90.21±1.72 <sup>a</sup>	90.00±1.00 <sup>a</sup>	89.60±1.40 <sup>a</sup>	89.51±1.49 <sup>a</sup>
Protein	12.40±0.47 <sup>b</sup>	13.00±0.65 <sup>b</sup>	11.57±0.43 <sup>a</sup>	11.87±0.67 <sup>a</sup>	14.57±0.33 <sup>°</sup>
Fat	5.88±0.21 <sup>b</sup>	7.43±0.11 <sup>b</sup>	5.63±0.00 <sup>b</sup>	6.92±0.32 <sup>b</sup>	4.02±0.21 <sup>a</sup>
Ash	1.12±0.00 <sup>a</sup>	1.38±0.00 <sup>a</sup>	1.52±0.00 <sup>a</sup>	1.67±0.00 <sup>a</sup>	3.23±0.03 <sup>b</sup>
Crude fibre	4.05±0.12 <sup>a</sup>	4.35±0.12 <sup>a</sup>	4.15±0.02 <sup>a</sup>	4.81±0.03 <sup>a</sup>	6.23±0.31 <sup>b</sup>
Carbohydrate	76.55±0.98 <sup>b</sup>	73.81±0.87 <sup>ab</sup>	77.14±0.54 <sup>b</sup>	74.73±0.34 <sup>ab</sup>	71.95±0.33 <sup>a</sup>

Table 1. Proximate composition of millet flours.\*

\* Values are on dry weight basis.

Table 2. Functional properties of sorghum flours.

Parameters	UMF	WSMF	LSMF	WCMF	LCMF
Water absorption capacity					
(g/g)	3.10±0.00 <sup>b</sup>	3.15±0.00 <sup>b</sup>	2.29±0.01 <sup>a</sup>	5.41±0.13 <sup>c</sup>	5.91±0.22 <sup>d</sup>
Oil absorption capacity (g/g)	1.22±0.00 <sup>a</sup>	1.52±0.11 <sup>°</sup>	1.43±0.00 <sup>b</sup>	1.58±0.22 <sup>c</sup>	1.21±0.00 <sup>a</sup>
Packed bulk density (g/cm <sup>3</sup> )	0.67±0.00 <sup>a</sup>	0.59±0.00 <sup>a</sup>	0.62±0.00 <sup>a</sup>	0.68±0.00 <sup>a</sup>	0.65±0.00 <sup>a</sup>
Loose bulk density (g/cm <sup>3</sup> )					
	0.50±0.01 <sup>a</sup>	0.50±0.01 <sup>a</sup>	0.51±0.02 <sup>a</sup>	0.51±0.02 <sup>a</sup>	0.53±0.02 <sup>a</sup>
pH	7.76±0.24 <sup>d</sup>	6.82±0.11 <sup>b</sup>	7.25±0.02 <sup>°</sup>	6.42±0.09 <sup>a</sup>	9.04±0.62 <sup>c</sup>
Emulsion capacity (%)					
	19.00±1.0 <sup>a</sup>	27.00±0.0 <sup>b</sup>	22.50±1.50 <sup>b</sup>	20.00±1.00 <sup>a</sup>	27.00±1.50 <sup>b</sup>
Emulsion stability (%)					
	20.00±0.0 <sup>a</sup>	23.50±1.00 <sup>b</sup>	20.00±1.50 <sup>a</sup>	20.00±0.00 <sup>a</sup>	26.00±1.00 <sup>b</sup>
Hygroscopicity (%)					
	30.00±2.00 <sup>a</sup>	30.00±1.00 <sup>a</sup>	38.00±0.00 <sup>b</sup>	38.00±1.5 <sup>b</sup>	41.00±0.00 <sup>c</sup>
Swelling power (ml/g)	2.17±0.01 <sup>a</sup>	3.00±0.13 <sup>b</sup>	4.36±0.00 <sup>c</sup>	4.36±0.00 <sup>c</sup>	4.50±0.02 <sup>d</sup>

Table 3. Anti-nutritional factors of sorghum flours.

Parameters	USF	WSSF	LSSF	WCSF	LCSF
Phytate (mg/100g)	89.64±0.41 <sup>a</sup>	73.50±1.36 <sup>b</sup>	60.32±1.32 <sup>c</sup>	46.10±0.51 <sup>d</sup>	59.03±0.97 <sup>°</sup>
Tannins (g/Kg)	1.91±0.90 <sup>ª</sup>	1.65±0.50 <sup>b</sup>	1.01±0.01 <sup>°</sup>	1.94±0.44 <sup>a</sup>	1.64±0.30 <sup>b</sup>
Trypsin inhibitor (mg/g)	9.16±0.26 <sup>b</sup>	9.05±0.20 <sup>b</sup>	6.64±0.00 <sup>c</sup>	13.40±1.00 <sup>a</sup>	5.31±0.31°
Cyanide (mg/100g)	14.20±0.00 <sup>b</sup>	7.80±0.88 <sup>d</sup>	10.50±0.50 <sup>°</sup>	16.05±0.00 <sup>a</sup>	11.14±0.14 <sup>c</sup>

Values are means and standard deviations of triplicate determination. Means in the same row not followed by the same superscript are significantly different (p<0.05).

#### Key:

UMF = Flour from untreated millet grains;

WSMF = Flour from millet grains soaked in water;

LSMF = Flour from millet grains soaked in lime solution;

WCMF =Flour from millet grains cooked in water;

LCMF = Flour from millet grains cooked in lime solution.