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Elaboration of Wine and Vinegar from Shea (Vitallera Paradoxa Gaernt) Fruit

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Abstract Shea pulp is considered a waste in the shea nut processing industry. An alcoholic beverage prepared by fermenting shea pulp with wine yeast was dry (12.95 ± 0.14%^{v}/v alcohol content), slightly acidic in taste (titratable acidity of 8.42± 0.42 g tartaric acidL^{-1} and pH of 3.42 ± 0.08) and had high phenolic content (3.21 ± 0.4gL^{-1}) with a distinct shea fruit smell and taste. Sensory evaluation rated the wine acceptable. GC-MS analysis revealed a total of 62aroma volatile compounds, with alcohols and esters being the most abundant groups. Identification and quantification of the compounds allowed a good characterization of the wine with fruity odour descriptors. Spontaneous acetic acid fermentation of the wine resulted in a decline in both alcohol and pH from 7.01 ± 0.04 %^{v}/v to 0.00 ± 0.04 %^{v}/v and 3.62 ± 0.03 to 2.71 ± 0.20 respectively over a period of 27 days. Volatile acidity however, increased from 0.1± 0.1 g acetic acidL^{-1} to 68.7 ± 0.3 g acetic acidL^{-1} over the same period. Processing of the shea pulp into wine and subsequently to vinegar is a realistic additional way of utilizing the shea pulp that hitherto go waste.

Keywords Shea pulp, wine, GC-MS, alcohols, esters, fruity

Introduction

The production of wine from fruits other than grapes has increased in recent years. Several other fruits however, have the potential for use in wine production. Over the years, fruit wines have been prepared from a variety of fruits, such as wild strawberry [1], orange [2], banana [3], bael [4], sour sop [5], cocoa [6], mango [7], pomegranate [8], and pineapple [9]. Vinegar, an important preservative and condiment with a variety of industrial, medical, and domestic uses is then obtained by fermentation of the wine, using acetic acid bacteria [10].

Wines and vinegars from grape, berry, apples and other pome fruits have traditionally come from Europe, Far East, Middle East, America, South and North Africa [11] but not from tropical countries like Ghana where these crops do not thrive. The need to identify traditional fruits that can serve as raw materials for the production of wines and vinegars cannot be overemphasized. One of such fruits is the shea fruit. The vast difference in fruit composition necessitates studies into production of these wines for an appropriate yeast strain, optimal temperature for fermentation and the type of treatment to be applied to the must of the fruit in the pre-fermentative phase and during the fermentation.

The shea tree (*Vitellaria paradoxa* Gaernt) grows wildly in all the three northern regions, the northern fringes of Ashanti, Brong-Ahafo, Eastern and Volta regions of Ghana. The tree produces fruits and nuts which are of high economic value to the locals and the nation. The Ghana Export Promotion Council estimated a modest $2.46billion

as earnings in 2013 from shea. Shea butter is extracted from the kernels of the fruit [12-14]. The shea fruit consists of a soft succulent pulp and a hard kernel surrounded by a shell. The pulp is very sweet and contains ascorbic acid, protein, a high content of tocopherol and mineral elements [15-20]. It acts as a food substitute during the dry season in Northern Ghana when there is food shortage.

Shea processing is mainly directed at the production of shea kernel and subsequently the butter. The fruit pulp constitutes over 30% of the total fruit weight, and large amounts of shea pulp are lost in the field after nut removal. By considering the size of shea plant population in the West African sub region, a lot of pulpis lost or underutilized. Attempts however were made to salvage the situation through the development of animal feed from the pulp in the past and now attention is being given to jams, brandy, gin, wine and vinegar. Considering the huge quantity of pulp that goes to waste, there is the need to explore alternative ways of utilizing the pulp. This study thus sought to evaluate the feasibility of preparing commercial-grade wine and vinegar from shea pulp.

Materials and Methods

Fruit Analysis

The fruits were analysed in relation to total weight, fruit:pulp and kernel:seed:pulp weight ratios. The pulp was characterised in relation to total soluble solids [21], total sugars [22], reducing sugars [23], total titratable acidity and pH, pectins [24] and phenolics [25]. Ash was estimated by incinerating 2 g of sample in a muffle furnace at 600 °C. Fat determination was done by extracting 5 g of sample with petroleum ether (40-60º C) in a Soxhlet apparatus.

Preparation of Must

Ripe undamaged fruits harvested from the Cocoa Research Institute of Ghana's Research station at Bole 9^0 01'N, $2^0$29'W, 309m above sea level in the Northern Savanna Zone of Ghana were used. Fruits were washed with clean water to remove any contaminant. Pulp was carefully separated from kernels with a knife to yield 20.5 kg per replicate. The fruits were partly crushed and water added at a ratio of 1:1.2. Pasteurized macerated extracts were ameliorated with sucrose to a density of 1.090in 30L drums (each drum containing 25 L of the must), covered tightly and allowed to cool. To each drum, 0.1g$L^{-1}$of sodium metabisulfite and 0.15 g$L^{-1}$of ammonium phosphate were added and stirred.

Preparation of Yeast Starter

Seven hundred and fifty millilitres of the must was used as the culture medium. Five grams of powdered commercial wine yeast, *Saccharomyces cerevisae* was dissolved in the must and allowed to stand for one hour at 28 ^oC.

Fermentation Process

Two hundred and fifty millilitres of the yeast starter was dispensed into each barrel containing the must and stirred gently. Each barrel was loosely closed and fermented at 22^oC. During fermentation, pH, titratable acidity, nonvolatile (fixed) acidity, volatile acidity, specific gravity and temperature were monitored daily until the specific gravity of the wines reached ≤1.000 and became stable. The wine was kept for a week before racking.

Clarification and Aging

Wine was racked (siphoned) into sterilized 23 L plastic containers and clarified with pectinase (Biocon) (0.02 gL^{-1}), bentonite (0.4 gL^{-1}), polyvinylpyrrolidone (0.18 gL^{-1}) and gelatin (0.09 gL^{-1}). The wine was tightly closed with mounted fermentation locks and allowed to stand for four weeks at a temperature of 17 ^oC. About 14 L of each wine was racked into clean 23 L capacity specially designed plastic containers for vinegar production and the rest racked into 4L conical flasks. The flasks were tightly closed with stoppers affixed with fermentation locks and placed in a dark room at a temperature of 17 ^oC and allowed to age.

Vinegar Containers

Three empty 23 L capacity gallons with two opposite rectangular openings of dimension 10 cm (length) x 5cm (height) created at the upper side of each gallon were used. For each gallon, one opening was covered with a plastic mesh and the opposite opening was covered with a transparent glass. The pores of the plastic mesh used in this experiment were small enough to prevent vinegar flies and other small insects from passing through.

Acetic acid Fermentation

Fourteen litres (14 L) of wine was racked from each wine barrel, four weeks after fining, into corresponding vinegar container. Each container of wine was diluted with distilled water to an alcohol concentration of 7%^{v}/v. Each container was covered with cotton wool and stored at room temperature in a well ventilated room. The wines were allowed to be naturally inoculated with Acetic acid bacteria (AAB) by air and insects (vinegar flies*). The mesh prevented the flies, which were drawn to the setup by the wines' fermentative smell*, from having direct contact with the wine but allowed the AAB associated with them to fall into the wine. The acetification process was daily monitored until all the ethanol was exhausted.

Bottling of Wine and Vinegar

Both wine and vinegar were centrifuged (MISTRAL 6000) at a speed of 4000 rpm at -5 ^{o}C for 25 minutes. Sodium metabisulfite (0.05 gL^{-1}) and potassium sorbate (0.18 gL^{-1}) were added to each container of wine after which they were filtered and pasteurised at a temperature of 68 ^{o}C for 10; minutes. The wine was bottled hot in dark green-coloured glass bottles (net content of 0.75L) which were sterilized in; 20.25% sodium metabisulfite solution and corked. The vinegars were bulked and diluted to an acetic acid concentration of about 4.5%^{v}/v. Sodium metabisulfite (0.05 gL^{-1}) and potassium sorbate (0.18 gL^{-1}) were added after which the vinegar was pasteurised at a temperature of 65^{o}C for 30 minutes. The vinegars were bottled in 250 mL white plastic bottles which were sterilized in 0.25% sodium metabisulfite solution.

Specific Gravity Determination

The specific gravity was measured according to AOAC method [21]. About 90 mL of the wine sample was placed in a transparent 100 mL glass cylinder and gently inverted five times, allowing gas to be given off each time the cylinder was held upright. The temperature of the sample was noted after which the specific gravity was measured with a glass hydrometer. The measured specific gravity was corrected using the appropriate temperature correction factor.

Alcohol Content Determination

Ethanol production during fermentation was monitored through the measurement of the specific gravity and the corresponding potential alcohol content was extrapolated from the relation:

$$\%\ \text{Potential alcohol by volume} = \frac{1000 \text{ x (initial specific gravity} - \text{Final specific gravity)}}{7.36}$$

The actual alcohol content of the finished wine and vinegar was determined by distillation as described by AOAC [21]. The test sample [100 mL) was diluted with 50mL of distilled water and the solution neutralized with 1M NaOH solution. The sample was then distilled at 100ºC until 100 mL of distillate was obtained. The percentage alcohol by volume was determined using an alcohol hydrometer.

pH and Titratable Acid Determination

The pH of both wine and vinegar was measured by using a pH meter (Mettler Toledo AG). The titratable acid was determined according to the method of AOAC [21] with slight modification. Carbon dioxide was first removed from the test samples by heating 25 mL of the sample to incipient boiling after which it was held 30s, swirled and cooled. Five millilitres degassed test portion was titrated with 0.1 M NaOH using 1%^{w}/v phenolphthalein as indicator. Titratable acid was calculated as g tartaric acidL^{-1} wine by using the formula:

$$\text{Titratable acid} = \frac{\text{milliliters of NaOH used x molarity of NaOH x 75}}{5}$$

Fixed and Volatile Acid Determination

The methods described by AOAC [21] were used in these determinations. Twenty-five millilitres of the test sample was carefully evaporated on a hot plate (Ikamag Reo) until the volume had reduced to 5-10 mL. Twenty-five millilitres of hot distilled water was added and the solutions again evaporated to a volume of 5-10 mL. The process was repeated two more times after which the residue was cooled and diluted to 50 mL with distilled water. This was titrated with 0.1 M NaOH using phenolphthalein as indicator. The fixed acid was expressed as g tartaric acid L^{-1}.The volatile acid was determined by subtracting the fixed acid value from the titratable acid value. The volatile acid was expressed as g acetic acid L^{-1}.

Assay of Total Phenolic Content

The phenolic content of the wine was determined by Folin-Ciocalteu's method [26-27]. Tannic acid was used as the standard phenolic compound. Each sample (0.1 mL) was added to 4.2 mL of distilled water and 0.5 mL of Folin-Ciocalteu reagent. After 1 minute of mixing, 1 mL of an 80% solution of sodium carbonate and 4.2 mL of distilled water were added. The mixture was left 2 h at room temperature in the dark and the absorbance at 760 nm was measured on a UV-VIS spectrophotometer (DU 730, Beckman Coulter). The total phenolic content was determined from the tannic acid calibration curve and the values expressed in g L^{-1} of wine.

Microbiological Analysis

Yeasts and moulds in the wine were enumerated using Rose Bengal Agar containing Dichloran and Cloramphenicol (DRBC Agar) (CONDA) and the plate count of bacteria was done using Casein-peptone Dextrose Yeast Agar (Plate Count Agar) (Fluka). Twenty milliliters of the wine samples were pipetted into 225 mL of Peptone Saline Diluent (Fluka). One millilitre of the solution was serially diluted up to 10^{-3} dilution. The spread plate technique was used during inoculation where 100 µL of the diluted samples were spread on the appropriate media. The Plate Count Agar and DRBC plates were incubated at 30 ^{o}C for 3 and 5 days respectively.

Sensory Evaluation Assay

Sensory attributes of the wine (clarity, colour, aroma, taste, astringency and aftertaste) were evaluated using a 5-point Hedonic scale (where 1 = dislike extremely and 5 = like extremely) according to Mohanty *et al* [28] method using 15 respondents.

Chemical Analysis

Substrates and Metabolites

Analyses for alcohols (methanol, ethanol and glycerol), organic acids (acetic acid, lactic acid, oxalic acid , malic acid, succinic acid, tartaric acid and citric acid) and carbohydrates (glucose, sucrose, fructose and mannitol) were carried out on a Waters liquid chromatography system comprising of a waters 1525 Binary pump with 2487 dual absorbance wavelength detector (UV) operated at 210nm and 2414 Refractive index detector. An ionic exchange column, Aminex HPX-87H (300 x 7.8mm, Bio-Rad Laboratories Ltd) coupled to a pre-column of cationic exchange (Bio-Rad Laboratories Ltd) was used to achieve chromatographic separation. Mobile phase was a 0.005 mol L^{-1} H_2SO_4 solution with flow-rate of 0.6 mL min^{-1}. The acids were detected both by U.V. absorbance (210 nm) and RI, while sugars and alcohols were by RI detection. Individual sugars, acids and alcohols were identified and quantified by comparison with retention times and from calibration curves of authentic standards respectively. All samples were examined in triplicate. The coefficient of variation was less than 5% in each case.

SPME procedure

Volatile components in the wine were extracted according to the method of Jiang and Zhang [29].
4-Nonanol used as internal standard was added to 5 mL of wine sample and 1g NaCl to a final concentration of 305 mg L^{-1} in a 15-mL vial. Tightly capped vial with PTFE septum was heated to 40°C for 30 minutes. A preconditioned SPME fiber (50/30-µm DVB/Carboxen/PDMS, supelco, Bellefonte, PA, USA) was inserted into the head space and extraction allowed to occur for 30min. Fibre was subsequently desorbed in the GC injector for 25 min.

GC-MS analysis
A Varian 3900 gas chromatograph possessing a septum-equipped temperature programmable injector (SPI), and an ion-trap mass spectrometer (Varian Saturn 2100T) was used. A capillary column (Factor Four VF-WaxMS Varian, 60 m x 0.25 mm i.d., 0.25 mm film thickness) with heliumas the carrier gas at a flow of l mL min^{-1} was used. The detector was operated in the electron-impact mode (70 eV), and mass spectra were acquired by scanning over the mass/charge (m/z) range of 29-360 with an acquisition rate of 610 ms. The temperature of the injector (SPI) was programmed to run from 20°C to 250°C at 18°C min^{-1} and was then maintained at 250°C during the analysis. The oven temperature was held at 60°C for 5 min, then programmed to run from 60°C to 220°C at 3°C min^{-1} and was finally maintained at 250°C for 25 min. Volatile compounds were identified using Varian Saturn GC/MS software (Version 5.2) by comparing mass spectra and linear retention indices with those of authentic standard compounds injected under the same conditions. 4-Nonanol was used as internal standard and added to each sample and standard to a final concentration of 305 mg L^{-1}. The quantification of the volatile compounds was expressed as 4-nonanol (internal standard) equivalents. The relative concentrations of the investigated compounds were calculated by relating the area of the internal standard to the area of the compound of interest.

Statistical Analysis
Values were expressed as mean ± standard deviation of three replications. The sensory evaluation data were presented as means of the panelist's score. Comparisons between scores were performed using Genstats (version 12.0) to determine statistical significance. The 0.05 level of probability was used as the criteria of significance in all instances.

Results and Discussion
Characterization of the pulp
The pulp of the shea fruits used in the wine production formed about 33% of the fresh fruit weight (Table 1) even though pulp percentages greater than 50 have been reported [30]. The shea fruit chemically characterized before initiating the fermentation process indicated an average pH value of 4.55, similar to that reported for banana [3] used in alcoholic beverage production. Total and reducing sugar concentrations were 16.1 % and 14.2 %, respectively. This makes the levels of sugars in the pulp comparable with other tropical fruits with potential for use in the beverage industry [31]. Total soluble solid concentration in pulp was quite high with 15.5◦Brix soluble solids which may not constitute only sugars for fermentation. Protein in the pulp was relatively high (7.24%). Proteinaceous materials are usually undesirable component of starting materials for wine production and were precipitated with bentonite. Sedimentation of the solids in the must was facilitated, aiding the decantation and filterability. Substrate contained about 8% pectin with less than 1% fat. The relatively high pectin content (4.42%) and low presence of endogenous pectinases necessitated the addition of pectinases to reduce viscosity of the pulp. Potassium level (850mg/100g FW) in the pulp was quite high compared to banana, a fruit recommended for its high potassium content (357 mg/100 g mean). Calcium, phosphorus, magnesium and zinc were also present at concentrations of 315.6, 40.8, 78.8 and 4.2 mg/100g FW, making it good sources of these minerals. Three main organic acids, comprising lactic, fumaric and oxalic were present in the shea pulp.

Table 1: Physico-chemical characteristics of shea fruit

Characteristics	Mean ± SE
Weight (g)	28.90 ± 4.01

Pulp (%)	33.6 ± 1.3
Nut (%)	66.4 ± 3.4
length (cm)	3.50 ± 0.35
diameter (cm)	2.50 ± 0.25
Total soluble solids (Brix)	15.5 ± 0.15
Total titratable acid (mEq L^{-1} in malic acid)	1.55 ± 0.02
pH	4.60 ± 0.04
Specific gravity of fruit extract	1.050
Lactic acid (mg Kg^{-1})	18.9 ± 3.2
Fumaric acid (mg Kg^{-1})	0.89 ± 0.02
Oxalic acid (mg Kg^{-1})	1.28 ± 0.20
Total sugars (%)	16.1 ± 0.11
Reducing sugars (%)	14.2 ± 0.16
Total pectin (%)	4.42 ± 1.10
Protein (%)	7.24 ± 1.02
Fat (%)	0.95 ± 0.14
Fibre	35.5 ± 0.50
Ash	4.10 ± 0.26
Phenolics (%)	0.65 ± 0.18
K mg/100 g FW	850.3 ± 9.8
P mg/100 g FW	40.9. ± 1.4
Ca mg/100 g FW	315.6 ± 3.6
Mg mg/100 g FW	78.8 ± 2.8
Zn mg/100 g FW	4.2 ± 0.8

FW – fresh weight

Shea grows in the wild in the West African sub-region with no commercial exploitation of the fruit. As with other tropical crops, alcoholic beverages likewines have been elaborated but not with shea. The specific gravity of the shea fruit extract was 1.050 before ameriolation. The primary fermentation process resulted in the decrease of specific gravity from 1.090 ± 0.01to a stable value of 0.990 ± 0.00 in day 21 (Table 2). Reduction to a level below 1.000 in the wine was indicative of all the fermentable sugars having been converted to ethanol by the yeast. Alcohol content increased from 0.0 % on day 1 to a value of12.85 ± 0.14 % at the end of the fermentation. Titritable and fixed acidity increased during the fermentation period from 3.1 ± 0.02 g tartaric acidL^{-1} to 8.4 ± 0.04 g tartaric acidL^{-1} and 3.7 ± 0.01 g tartaric acidL^{-1} to 7.2 ± 0.01 g tartaric acidL^{-1} respectively. Titratable acidity of wines generally range between 0.5 % and 1.0 % [32] and that of the shea winefell within this limit. Concentration and type of organic acids present in a particular fruit used for wine production influences the fixed acidity [33]. Generally, the shea wine appeared more acidic than the must. pH decreased from 4.55in must to 3.42 in the wine at the end of fermentation. The pH of the shea wine is comparable to that of other wines [34]. Low pH is reported to give fermenting yeasts competitive advantage in natural environments [35]. Volatile acidity increased to 0.31 ± 0.02 g acetic acidL^{-1} during the fermentation, probably due to the activity of Acetic acid bacteria (AAB) and other bacteria which, being ubiquitous in nature, were able to convert some of the alcohols into volatile acids (mostly acetic acid) [36]. AAB's continuous activity was however suppressed as the fermentation progressed with the alcohol level going beyond their 7 to 9% tolerance level [37].

Table 2: Physicochemical characteristics of Shea must and wine

Parameters	Shea must	Shea wine
Specific gravity	1.090 ± 0.00	0.990 ± 0.00
Alcohol (%)	0.00 ± 0.00	12.95 ± 0.14
pH	4.55 ± 0.02	3.42 ± 0.08
Titratable acidity (g tartaric acid L^{-1})	3.1 ± 0.02	8.42 ± 0.42
Fixed acidity (g tartaric acid L^{-1})	3.7 ± 0.01	7.2 ± 0.01
Volatile acidity (g acetic acid L^{-1})	0.1 ± 0.01	0.31 ± 0.01
Total phenolic content (g L^{-1})	4.10 ± 0.12	3.21 ± 0.4

Values are means ± SD of three determinations

The total phenolic content decreased from an initial level of 4.10 ± 0.12 gL^{-1} in the must to a final level of 3.21 ± 0.gL^{-1} in the wine (Table 2). This disparity may be as a result of the activity of the fining agents that were added to the wine. Mohanty *et al.* [28] similarly observed a decrease in both total phenolic (g 100 mL^{-1}) and tannin (mg 100 mL^{-1}) contents from 0.13 ± 0.01 and 2.2 ± 0.13 respectively in shea must to 0.12 ± 0.03 and 1.9 ± 0.22, respectively in shea wine. Phenolic compounds, known to be antioxidants, are capable of protecting cell membranes from free-radical mediated oxidative damage which has been implicated in diverse pathological conditions [38]. Thus the presence of these compounds in shea wine may be beneficial.

The absence of microbial growth on both the Rose Bengal Agar containing Dichloran and Cloramphenicol (DRBC) and Plate Count Agar (Casein-peptone Dextrose Yeast Agar) indicated that the wine fermented and aged without any microbial spoilage. This may be due to the high alcohol content (12.58 ± 0.24%) of the wine which inhibited the growth of the microorganisms [38]. The absence of sugars in the wine also served as an annihilation factor for some of the organisms [39].

The sensory evaluation of the shea wine showed that, the panelists had likeness for the wine in terms of clarity, colour, taste, astringency and aftertaste (Table 4). However, the aroma was not too acceptable possibly because most of them were not familiar with the natural shea fruit smell.

Table 4: Sensory evaluation of the Shea wine

Attributes *	Shea wine
Clarity	4.1 ± 0.5
Colour	3.0 ± 0.4
Aroma	2.5 ± 0.6
Taste	3.6 ± 0.3
Astringency	3.8 ± 0.6
Aftertaste	4.2 ± 0.7

Values are means ± SD of the panelists' scores. *n* = 15.

*1= dislike extremely; 2 = like moderately; 3 = like much; 4 = like very much; 5 = like extremely

Organic acids, glycerol, ethanol and sugars

Organic acids differ in concentration and type from fruit to fruit and are also influenced by the degree of ripening of the fruit [39]. Seven acids were identified in the wine, three of them (Fumaric, Oxalic and Tartaric) originally present in the fruit (Table 5). Acids influencing and contributing to the acidity and quality of wines mainly include tartaric, malic, citric, lactic and succinic acids[40].Ethanol formed the main product of the alcoholic fermentation by yeast, concentrations depending on the initial total sugar concentration in the must. Glycerol, a product of alcoholic fermentation wasthe second major alcohol formed in the wine. It is reported to impart sweetness to wines [40]

Table 5: Concentration (g L^{-1}) of residual sugars, organic acids and alcohols in the fermented shea pulp beverage as determined by HPLC

Compounds	Concentration in shea wine (gL^{-1})

Carbohydrates	
Glucose	0.51± 0.10
Fructose	n.d
Mannitol	0. 47 ± 0.05
Alcohols	
Ethanol	58.6 ± 1.7
Glycerol	5.5 ± 0.51
Methanol	Trace
Acids	
Acetic acid	0.09 ± 0.02
Lactic	5.05 ± 0.17
Malic	1.55 ± 0.7
Oxalic	1.2 ± 0.03
Succinic	1.0 ± 0.27
Tartaric	0.4 ± 0.02
Fumaric	0.14 ± 0.03

n.d – not detected

Volatile aroma compounds

Alcohols (both aliphatic and aromatic) formed the second largest group of aroma compounds identified as a result of the degradation of carbohydrates, lipids and amino acids [54]. Aside ethanol, the main component of wines, 2-Phenylethanol was the most abundant among the 16 alcohols identified but all had levels lower than their reported flavour threshold concentrations (Table 6).

Esters, especially ethyl esters contribute immensely to the aroma of wines. They tend to contribute floral and fruity sensory properties of wines [55]. Among the 15 esters present, 12 were ethyl esters and 3 acetates. Seven (Ethyl butanoate, ethyl propionate, ethyl-2-methylbutanoate, ethyl-3-methylbutanoate, ethyl hexanoate, Ethyl dodecanoate and 2-Phenylethyl acetate) were present beyond their flavor threshold values (Table 6). These can be considered as contributing immensely to the aroma of the wine. Whereas ethyl esters of fatty acids are produced during alcoholic fermentation, the acetate esters result from the reaction of acetyl-CoA with higher alcohols formed during the degradation of amino acids or carbohydrates [55].

Fermentation condition and composition of must greatly influences the production of fatty acids [56]. These acids enzymatically formed during fermentation can contribute fruity, cheese, fatty, and rancid notes to the wine's sensory properties [57]. Among the ten fatty acids quantified, only hexanoic acid exceeded the threshold concentration. It is known to impart rancid and cheesy aroma to wine. The other fatty acids reportedly at low levels of concentrations between 4 to 10mgL^{-1} positively impart pleasant aroma to wine [58] and hence suggested to impart on the aroma of the shea wine since their concentrations fall within the range.

Of the seven carbonyl compounds identified in the wine, six were aldehydes and one ketone. Two (ethanal and nonanal) appeared at concentrations higher than their odour thresholds. Ethanal forms part of almost every fruit wine as a result of yeast metabolism. It imparts a fruity pleasant aroma at low concentrations but irritating and pungent odor at high levels [59]. The levels observed in the wine exceeded the threshold value and likely to impart on the flavor of the wine.

Terpenes being secondary plant constituents are associated with floral and citric aromas and reported to have a very low olfactory threshold [41]. Their presence in wines has hardly been reported but known not to be changed by yeast metabolism [60]. Low concentrations of four terpenes were detected in the wine. They were linalool, a-terpineol, citronellol and geraniol, forming less than 0.1% of the total volatile compounds. Their contribution to the shea wine would seem negligible when their odor thresholds are considered.

Four ɣ-lactones were identified with the most abundant being ϒ-butyrolactone. Fermented products tend to contain ϒ-butyrolactone probably arising from glutamic acid or related compounds [61]. Only 4-(1-Hydroxyethyl)-ϒ-

butyrolactone exceeded the reported odour threshold value. The lactones however formed 6.1% of the total concentration of aroma compounds.

Table 6: GC-MS of quantified volatile compounds in shea wine, their odor threshold and descriptors as reported in literature

	Threshold Conc (µg L^{-1})	**Odor Descriptor**	**Conc (µg L^{-1})**
Alcohols (15)			
1-Hexanol	8,000[41]	Green, grass	2500.4 ± 22.1
(*Z*)-3-hexen-1-ol	400[41]	Green	52.9 ± 0.7
(*E*)-3-hexen-1-ol	400[41]	Green, floral	102.2 ± 0.4
(*E*)-2-hexen-1-ol	400[42]	Green, grass, herb	89.5 ± 2.1
(*Z*)-2-hexen-1-ol	400[42]	Green, grass, herb	22.7 ± 1.2
1-Butanol	150,000[43]	Medicinal, alcohol	1001.3 ± 30.7
4-Methyl-1-pentanol	50,000[44]	-	8.9 ± 0.3
2-Heptanol	70[45]	Fruity, mouldy, musty	35.5 ± 0.9
3-Methyl-2-buten-1-ol	NA		4.8 ± 0.8
3-Methyl-1-pentanol	NA		7.5 ± 1.1
2-Ethyl-1-hexanol	8,000[46]	Mushroom, sweet fruity	7000.6 ± 50.7
1-Octanol	120[41]	Intense citrus, roses	43.4 ± 0.7
Furfurol	1,000[47]	Mouldy hay	22.1 ± 0.9
2-Phenylethanol	14,000[41]	Flowery, pollen, perfumed	8163.7 ± 15.7
Phenylmethanol	200,000[48]	Citrusy, sweet	50.1 ± 0.8
Subtotal (µg L^{-1})			19105.6
Subtotal (%)			11.1
Ethyl esters (13)			
Ethyl propionate	45[41]	Fruity	56.9 ± 0.4
Ethyl butanoate	20[41]	Fruity, papaya, butter, sweetish, apple, perfumed	98.4 ± 1.7
Ethyl 2-methylbutanoate	18[49]	Fruity, sweet fruity	40.3 ± 1.1
Ethyl 3-methylbutanoate	3[49]	Fruity, berry-like, Sweet fruity	11.3 ± 0.2
Ethyl hexanoate	5, 14[47,49]	Fruity, anise	22.6 ± 0.7
Ethyl -2-hydroxypropanoate	154,636[43]	Lactic, raspberry	11522.2 ± 55.8
Ethyl octanoate	2[41]	Pineapple, pear, floral	0.5 ± 0.1
Ethyl dodecanoate	1500[42]	Flowery, fruity	1557. 6 ± 2.9
Diethyl succinate	500,000[43]	Light fruity	23850 ± 24.1
Diethyl glutarate	NA		1.2 ± 0.1
Diethyl malate	NA		36.8 ± 1.1
Mono-ethyl succinate)	NA	sweat, sour, fruity	319.5 ± 0.7
Subtotal (µg L^{-1})			37,517.3
Subtotal (%)			21.8
Acetates (3)			

3-Methylbutyl acetate	30 [41]	Banana, fruity	7.1 ± 0.5
Ethylphenyl acetate	NA		5.7 ± 0.3
2-Phenylethyl acetate	250 [41]	Pleasant, flowery	311.2 ± 1.9
Subtotal (μg L^{-1})			324
Subtotal (%)			0.2
Monoterpenic alcohols (4)			
Linalool	25.2 [49]	Fruity, citric	22.5 ± 0.8
a-Terpineol	250 [49]	Pine, terpenoids	215.8 ± 1.7
Citronellol	100 [41]	Green lemon	14.6 ± 0.9
Geraniol	NA	Rose-like, citrus-like	1.8 ± 0.2
Subtotal (μg L^{-1})			254.7
Subtotal (%)			0.1
Volatile phenols (4)			
Methyl salicylate	NA		1.2 ± 0.5
4-Vinylguaiacol	21 [50]	Clove-like	5.6 ± 0.3
Vanillin	65 [51]	Vanilla-like, sweet, vanilla	11.2 ± 0.6
3,4,5-Trimethoxyphenol	NA		4.7 ± 0.2
Subtotal (μg L^{-1})			22.7
Subtotal (%)			0.0
Acids (10)			
Ethanoic acid	200,000 [41]	Acid, fatty	90,000 ± 40.2
Propanoic acid	8,100 [48]	Vinegarish	10.5 ± 0.6
2-methylpropanoic acid	200,000 [41]	bitter, fatty, sweaty	67.4 ± 0.7
Butanoic acid	173 [49]	Sweaty, Cheese, rancid	34.7 ± 1.4
Hexanoic acid	3,000 [41]	Cheese, rancid, fatty	3460 ± 4.7
Heptanoic acid	3,000 [52]	Fatty, dry	3.2 ± 0.5
Octanoic acid	500 [49]	Rancid, harsh, cheese, fatty acid, vegetable oil	242 ± 2.7
Decanoic acid	15,000 [41]	Fatty, unpleasant, soap, wax, tallow	6002 ± 30.6
Tetradecanoic acid	NA		5.9 ± 0.3
Hexadecanoic acid	NA		72.1 ± 2.2
Subtotal (μg L^{-1})			99,898
Subtotal (%)			58.0
Carbonyl compounds (7)			
Ethanal	25 [50]	Fresh, green	30.1 ± 1.9
3-Hydroxybutanone	150000 [41,52]	Flowery, wet	2550 ± 30.7
Octanal	3.4 [50]	Citrus-like, Green	3.2 ± 0.2
Nonanal	2.8 [50]	Green, slightly pungent	3.6 ± 0.7
2-Furaldehyde	8000 [51]	Almonds	40.8 ± 0.5
Benzaldehyde	2000 [53]	Almond like odor	548.5 ± 10.6
Decanal	1000 [44]	Grassy, orange skin-like	750.3 ± 2.7

Subtotal (µg L^{-1})			3926.5
Subtotal (%)			2.3
Sulfur (3)			
3-(Methylthio)-1-propanol	500 [41]	Boiled potato, rubber	695.8 ± 5.9
2-Methyltetrahydrothiofeno-3-one	NA		19.6 ± 0.1
Benzothiazole	NA		11.9 ± 1.1
Subtotal (µg L^{-1})			727.3
Subtotal (%)			0.4
Lactones (4)			
ϒ-Butyrolactone	NA	Sweet buttery	7230 ± 50.9
5-Oxo-ϒ-hexalactone	1600	Alcoholic	460 ± 5.6
4-(1-Hydroxyethyl)-ϒ-butyrolactone	1600	Red fruits	2534 ± 12.8
Tetrahydro-4-hydroxy-4-methyl-2*H*-pyran-2-one	NA	Caramel	257 ± 6.7
Subtotal (µg L^{-1})			10481
Subtotal (%)			6.1
Total			172256.9

NA- threshold values not available; Numbers in brackets indicates references.

Vinegar

The conversion of the alcohol in the wine produced to acetic acid by the activity of AAB resulted in the formation of vinegar. pH decreased from 4.23 ± 0.03 to 2.40 ± 0.27 as alcohol content decreased from 7.14 ± 0.04 % to 0.00 ± 0.04 % at the end of the acetic acid fermentation. The volatile acidity however increased from 0.10 ± 0.01 g acetic acid L^{-1} to 69.9 ± 0.3 g acetic acid L^{-1} on the 27th day of fermentation. The end of the fermentation was signaled by the over-oxidation which occurred after the 27th day of fermentation leading to a decrease in volatile acid content to 68.5 ± 0.3 g acetic acid L^{-1} on the 29th day. Some strains of AAB are known to be over-oxidizers and tend to convert acetic acid to carbon dioxide and water in the absence of ethanol [62].

Generally, the properties of the vinegar produced in this study were similar to standard vinegar [59]. Though the acetic acid content (measured as volatile acid) was slightly higher than 4.5% as stated by Raji *et al.* [63] (Table 5), it fell within the range required by most countries [63].

Conclusion

A total of 62 compounds were identified and quantified in the wine. The presence of higher levels of higher alcohols, esters and fatty acids characterized the wine. According to the Odor Activity values (OAVs), 11 volatile compounds were present in the wine at concentrations higher than their threshold values, but ethyl butanoate, ethyl propionate, ethyl-2-methylbutanoate, ethyl-3-methylbutanoate, ethyl hexanoate, Ethyl dodecanoate and 2-Phenylethyl acetate were the most characteristic aroma-active compounds of the wine. Spontaneous fermentation method, which requires no complex or expensive equipment, was used to produce quality shea vinegar with attributes which conform to generally accepted standards. Commercial grade wine and vinegar production often requires the use sophisticated fermentation tanks which are often too expensive for the average person in a developing country to acquire. This study shows that ordinary household materials could be used to commercially exploit the underutilized shea pulp in Ghana through the production of wine and vinegar.

Conflicts of Interest

No potential conflicts of interest exist.

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