Bengtsson, N. E., Jakobsson, B., and Dagerskog, M. (1976). Cooking of beef by oven roasting: A study of heat and mass transfer. *Journal of Food Science, 41, 1047–1053*.
Brown, A. (2004). Understanding food: Principles and preparation: 2nd Edition. Thomson Warsworth, Belmont
FAO, (2007) Small-Scale Sausage production, Food and Agricultue Organization of the United Nation, Rome. ISBN 92-5-102187-2.
Laakkonen, E. (1973). Factors effecting tenderness during heating of meat. *Advances in Food Research* 20: 257-323
Offer, G., and Knight, P. (1988). In R. A. Lawrie (Ed.), The structural basis of water holding in meat. Part 1: General principles and water uptake in meat processing. *Developments in Meat Science, 4. (pp. 63–171). London: Elsevier Applied Science*
Vittadini, E., Rinaldi, M., Chiavaro, E., Barbanti, D., and Massini, R. (2005). The effect of different convection cooking methods on the instrumental quality and yield of pork longissimus dorsi. *Meat Science*, 69, 749−756.

CHEMICAL PROFILE OF LIQUID SMOKE GENERATED FROM *PARKIA BIGLABOSA VITELLERIA PARADOXA AND EUCALYPTUS GLOBULUS* WOOD USING GC-MS ANALYSIS

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ABSTRACT
This research was conducted to compare the chemical profile of liquid smoke generated from *Parkia biglabosa*, *Vitelleria paradoxa* and *Eucalyptus globulus* using GCMS Analysis. The results of the liquid smoke analysis as indicated by Gas Chromatography and Mass Spectrometry (GCMS) identified a total of 22, 18, and 15 chemical compounds in Liquid smoke parkia (LSP), Liquid smoke sheabutter (LSS) and Liquid smoke eucalyptus (LSE), respectively. The major components found across the liquid smokes produced included 2,4-Di-tert-butylphenol; 2,6 dimethoxy phenols; 3,5-dimethoxy-4-hydroxytoluene; 5-tert-butylpyrogallol and 2-Propanone, 1-(4-hydroxy-3-methoxyphenyl).
Key words: Liquid Smoke, GC-MS analysis chemical profile, Chemical compounds, woods.

INTRODUCTION
The antimicrobial, colour and flavour properties of wood smoke are due to the presence of phenolic, carbonylic and acidic compounds naturally present in wood (Holley and Patel, 2005). The preservative action of the smoking process for foods depends upon elements such as processing temperature and partial dehydration. The conventional smoking process has been continuously substituted by the use of smoke flavourings like liquid smoke (Guillen and Ibargoitia, 1998). According to the International Organization of the Flavour Industry (IOFI), liquid smoke is a complex mixtures of components of smoke obtained by heating untreated wood to pyrolysis by a limited and controlled amount of air, dry distillation or superheated steam, then collecting the wood smoke in an aqueous collection system (IOFI, 2012).
Liquid smoke extracts has the capability to flavour and impart characteristic smoke colour to food products (Gary, 1992). Liquid smoke is known to possess antioxidant properties, and serve as natural alternatives to conventional antimicrobials (VanLoo *et al.*, 2012). It has been demonstrated in previous studies that liquid smoke preparation are effective against various types of spoilage microorganisms (Yaqoob,2022; Milly *et al*., 2005). It exhibits antimicrobial activity against *Listeria, Escherichia coli, Staphylococcus aureus,* and *Staphylicocal enterotoxins* which are the main contaminants of meat products (Lingbeck *et al*., 2014). The antimicrobial properties, colour and flavour of wood smoke are due to the presence of phenolic and carbonylic compounds naturally present in wood (Holley and Patel, 2005).
The carbonization of many different types of wood can be used to produce various wood vinegars including Eucalyptus, oak, bamboo, mangrove, coconut shell, and apple trees. (Yang *et al.*, 2016). The chemical composition of liquid smoke depends primarily on the wood type (soft or hard) and moisture

content of wood, the latter influences the pyrolysis temperature and duration of smoke generation (Cadwallader, 2007).

Though many studies have indicated that liquid smoke has potential as a natural antioxidant and antimicrobial agent, however, previous studies also reported that wood vinegar produced using different source materials might present different types and levels of chemical constituents. As reported by Alexandre *et al*. (2020), the commercial producers of liquid smoke don't disclose the specification of the chemical composition of liquid smoke produced as it is the industries property right and a patent information. This makes it difficult to determine the appropriate level and types of liquid smoke to be added to food (meat). Hence, establishing the chemical composition of any liquid smoke is the major step to better understand the interaction with the chemical components of food systems. This may also be advantageous in predicting the resulting sensorial properties of the final meat product. This study investigates the important chemical composition of full strength liquid smoke from *Pakia biglabosa*, *Vitelleria paradoxa* and *Eucalyptus globulus* using GC-MS analysis.

Gas chromatography mass spectroscopy (GC-MS), a sensitive hyphenated system, is widely used to analyze, identify, and quantify the chemical compositions of natural products. The unknown chemical composition of wood vinegar from different plants can be determined by matching of the peak mass spectra distribution with the NIST MS database (Yang *et al.*, 2016)

MATERIALS AND METHODS

Experimental Materials

The materials used included; locally fabricated liquid smoke condenser, wood chips gotten from *Parkia biglobosa* (Pakia), *Vitelleria paradoxa* (Shea butter) *and Eucalyptus globulus* (Eucalyptus),

Liquid Smoke Production

The different wood chips were sorted, labeled and soaked in water at room temperature for 30 minutes prior to pyrolysis. The individual wood chip variety were placed in different liquid smoke condenser. The liquid smoke formed thereafter was collected and allowed to stand for 48 hours so as to allow the tars and sediments to settle at the bottom of the cup. It was later filtered with a paper strainer and was stored in labeled plastic bottles for subsequent use. Samples of the individual liquid smoke were taken to the Multi-User Research Laboratory at the Chemistry Department , Ahmadu Bello University/ Zaria for their chemical profile using the GSMS machine.

Procedure for Liquid-Liquid Extraction for the GCMS Analysis

The individual liquid smoke where subjected to liquid-liquid extraction using the procedure as described by Hale (2017) with little modifications. The liquid smoke was extracted using an immiscible solvent, chloroform (GCMS grade). Thirty (30) mls of the individual liquid smoke were measured and was added to twice the volume of chloroform (60mls) in a conical flask. The samples were mixed together for 90 minutes in an ultra-sonicator (uses vibration and sound waves to mix samples together). Thereafter, it was allowed to stand for 30min so as to allow it split. The layers formed were separated using a separatory funnel where the chloroform extracted liquid smoke was collected. Two (2) mls of the chloroform extracted liquid smoke was measured and was diluted with chloroform to a ratio of 1:10. 1 µL of the mixture was then measured into the GCMS vials for analysis.

GCMS analysis

The liquid smoke chloroforme extracts were subjected to GCMS analysis using an Agilent system consisting of a Gas Chromatoghrapher (model 7890B) and a Mass Spectrometer detector (model 5977A). The carrier gas was helium (99.99%) with a flow rate of 1mls/min. The injector and detector temperatures were respectively set at 250^0C and 25^0C. Spectra were obtained over a scan range of 46 to 600 amu at 2 scans/s. The GC program was set as follows: the initial temperature was 60^0C and held for 10 min, then increased by 2^0C/min to 80^0C and held for 5 min, then raised by 2^0C/min to 110^0C and held 6 min, then raised by 2^0C/min to 120^0C and held for 5 min, and finally raised by 5^0C/min to 180^0C and held at 180^0C for 2 min. The chloroform extracted liquid smoke (1.0µL) was injected automatically while maintaining a solvent delay of 4 min. The total run time was 36 minutes. Interpretation of the mass spectrum was made by comparing the peak distribution against the database of National Institute Standard and Technology (NIST/MS 14.0, Gaithersburg, MD, USA). Relative percentages of the chemical compositions were calculated based on the GC peak areas without correction.

Results and Discussion

Table 1: Chemical compounds found in three different liquid smoke

Names of Compounds	Peak Area (%)		
	LSP	LSS	LSE
Phenol, 4-ethyl-2-methoxy-	-	1.57	-
Phenol, 2,6-dimethoxy-*	4.81	12.56	19.85
Vanillin	-	2.26	
Tridecane*	0.69	2.12	3.15
1-Tetradecene*	0.90	2.21	2.48
Tetradecane*	1.08	2.11	2.28
Naphthalene, 1-methyl-	-	1.68	
Naphthalene, 2-dimethyl-	0.76	-	-
3,5-Dimethoxy-4-hydroxytoluene*	2.05	9.45	10.71
4-hydroxy-2-methoxybenaldehyde	-		1.74
Pentadecane	0.36	0.69	-
2,4-Di-tert-butylphenol*	10.51	19.72	18.68
5-tert-butylpyrogallol	-	4.62	7.22
2-Propanone, 1-(4-hydroxy-3-methoxyphenyl)-*	1.22	2.73	5.03
Ethanone, 1-(4-hydroxy-3,5-dimethoxyphenyl)-	-	-	1.89
Cetene*	1.67	3.06	3.01
3,5-Dimethoxy-4-hydroxyphenylacetic acid	1.07	3.43	-
Hexadecanoic acid, methyl ester	1.03	-	-
Dibutyl phthalate*	2.34	2.79	2.15
n-Hexadecanoic acid	1.03	-	-
cis-13-Octadecenoic acid, methyl ester	5.65	-	-
trans-13-Octadecenoic acid	0.92		
2-Methyl-Z,Z-3,13-octadecadienol	1.39		
beta.-Amyrin	0.31		
3-Eicosine, (E)-	-	3.84	2.97
1-Octadecene*	1.90	4.25	3.99
1-Docosene*	1.64	2.56	2.54
1-Tetracosene	0.80	-	-
Bis(2-ethylhexyl) phthalate	2.75	-	-

LSP- Liquid Smoke Parkia, LSS-Liquid Smoke Sheabutter, LSE- liquid Smoke Eucalyptus

Chemical Compounds found in the Individual Liquid Smokes Produced

The chemical compound of a liquid smoke varies based on their specific characteristics, although the major component are usually groups of phenols, acids, aldehydes, ketones, furans, etc. (Swastawati, 2008). More than 200 chemical constituents of liquid smoke have been identified from different wood resources (Yang *et al*., 2016).

The results from this study revealed a total number of 22, 18 and 15 compounds in LSP, LSS and LSE respectively. These compounds have similarities ranging from 68-99% with those in the GCMS library.

From the result (Table 1) obtained in this study, LSP had the most compounds identified but LSE's compounds had the highest concentrations in terms of the peak areas of the compounds present in it. The major compounds found in LSP were; 2, 4-Di-tert-butylphenol (10.51%), cis-13-Octadecenoic acid, methyl ester (5.56), 2,6 dimethoxy phenols also known as syringol (5.81%) and 3,5-dimethoxy-4-hydroxytoluene (2.05%). Followed by LSS whose major components were; 2,4-Di-tert-butylphenol (19.72%), 2,6 dimethoxy phenols (12.56%), 3,5-dimethoxy-4-hydroxytoluene (9.45%) and 5-tert-butylpyrogallol (4.62%) and the least was in LSE with 2,4-Di-tert-butylphenol (18.68%), 2,6 dimethoxy phenols (19.85%), 3,5-dimethoxy-4-hydroxytoluene (10.71 %) , 5-tert-butylpyrogallol (7.22%) and 2-Propanone, 1-(4-hydroxy-3-methoxyphenyl)- (5.03%) as its major compounds.

Most of the compounds identified (Table 1) are the same across all the treatments although there were variations in their peak area and retention time. The peak areas indicated that acids, carbonyls, phenols, and organic acids were present in the condensed smoke across all the treatments. The compared properties of liquid smoke produced from Parkia, Sheabutter and Eucalyptus showed no much variations in their chemical composition and quality though, their retention time and peak area were different which means their concentrations are not the same this is attributed to the fact that different plant materials were used in producing the liquid smoke.

Some of the chemical compounds even though similar to the findings of Alexandre *et al*. (2020), Montazeri (2013), Toledo (2008) and Milly *et al*. (2005), still varied widely in terms of their retention time and peak areas. The differences observed in the presence and absence of some compounds across the treatments can be attributed to the variations in the type of wood used, the moisture content, and the rate of aeration during smoke generation. This is supported by the findings of Toledo (2008) who reported that not all published data on smoke composition reported by different authors have the same compounds. Therefore, the concentration of the different compounds and types varied among the treatments.

CONCLUSION

From the findings of this study, the chemical profile and composition of the liquid smokes varied across the different liquid smokes generated with LSP having the highest number of compounds identified.

REFERENCES

Alexandre Santos Pimenta, Maíra Fasciotti, Thays Vieira Monteiro & Elias Costa de Souza (2020): Chemical profiling of liposoluble liquid smokes obtained from Eucalyptuswood tar: confirmation of absence of polycyclic aromatic hydrocarbons, Food Additives &Contaminants: Part A, DOI:10.1080/19440049.2020.1740337

Cadwallader, K. R. (2007). Wood smoke flavour. In L. M. L. Nollet, (ed). *Handbook of Meat,_Poultry and Seafood Quality*. Blackwell Publishing, Ames, IA. Pp. 201–210.

Gary, L. U. (1992). High browning liquid smoke composition and method of making a high browning liquid smoke composition. U.S. Patent 5135770.

Guillen, M. D. and Ibargoitia, M. L. (1998). New components with potential antioxidant and organoleptic properties, detected for the first time in liquid smoke flavouring preparations. *Journal of Agricultural and Food Chemistry*, 46: 1276-1285.

Hale, S. C. (2017). Effectiveness of Liquid-Liquid Extraction, Solid Phase Extraction, and Headspace Technique for Determination of Some Volatile Water-Soluble Compounds of Rose Aromatic Water, *International Journal of Analytical Chemistry*, vol. 2017, Article ID 4870671, 7 pages, 2017. https://doi.org/10.1155/2017/4870671.

Holley, R. A. and Patel, D. (2005).Improvement in shelf-life and safety of perishable foods by plant essential oils and smoke antimicrobials. *Food Microbiology*,22(4): 273–292.

International Organization of the Flavour Industry (IOFI) guidelines for the preparation of smoke flavourings. *Section 15 of IOFI Code of Practice*. Update version 1.3, 2012.

Lingbeck, J. M., Cordero P., O'Bryan, C. A., Johnson, M.G., Ricke, S. C. and Crandall, P. G. (2014). Functionality of liquid smoke as an all-natural antimicrobial in food. *Journal of Meat Science*, 97(2):197-206. Doi: 10.1016.

Milly, P. J., Toledo, R. T. and Ramakrishnan, S. (2005). Determination of minimum inhibitory concentrations of liquid smoke fractions. *Journal of Food Science*, 70:M12–M17.

Montazeri, N., Oliveira, A., Himelbloom, B., Leigh, M. and Crapo, C. (2013). Chemical characterization of commercial liquid smoke products. *Journal of Food science and nutrition*,1:102-115.

Swastawati, F. (2008). Quality and safety of smoked catfish (*Aries talassinus*) using paddy chaff and coconut shell liquid smoke. *Journal of Coastal Development,* 12(1) : 47- 55.

Toledo, R.T. (2008). Wood Smoke Components and Functional Properties. In: Kramer, D. E. and Brown, L. (eds). *International smoked seafood conference proceedings*. Alaska Sea Grant College Program, Anchorage, AK. Pp. 55–61. doi:10.4027/isscp.2008.12

Vanloo, E., Babu, D., Crandall, P. and Ricke, S. (2012). Screening of commercial and pecan shell extracted liquid smoke agent as natural antimicrobial against food borne pathogen. *Journal of Food Protection*, 75: 1148-52.

Yang, J.F., Yang, C.H., Liang, M.T., Gao, Z.J., Wu, Y.W., and Chuang, L.Y. (2016). Chemical Composition, Antioxidant, and Antibacterial Activity of Wood Vinegar from Litchi chinensis. *Molecules* 21:1150. doi:10.3390/molecules21091150.

Yaqoob, R., Yaqoob, U., Ahmad, Y., Muhammad H., Shehu, F. N., Adamu, H**.** (2022). Comparative Evaluation of Liquid Smoke Made From *Parkia biglabosa Vitelleria paradoxa and Eucalyptus globulus* on the Microbial Properties of Grilled Broiler Chicken Meat. *47th Annual Conference of the Nigerian Society for Animal production (Jos,2020), University of Jos- Jos*. March, 2022.