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Drying kinetics and impact on the volatile compounds of ogi using response surface modelling

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Abstract

This research aimed at studying the drying kinetic and impact on the volatile compounds of ogi slurry dried within designed drying condition (Drying temperature: 40, 50 and 60°C and drying time: 8, 10 and 12 hrs) using a central composite rotatable experimental design with two factors and five levels (-1.4142, -1, 0, 1 and 1.4142). Gas Chromatography-Mass Spectrometry (GC-MS) was used to detect volatile compounds of the dried ogi. The results revealed that the moisture content and moisture ratio of the samples decreased continually with the increase in drying temperature and time. The drying rate of the operation also decreased with increased drying time. The retention time of thirty-five volatile compounds detected in fresh ogi, dried ogi and dried maize grains ranged from 9.67 to 26.96 mins. There was a significant difference ($p<0.05$) between the concentration of volatile compounds detected in wet ogi compared with dried ogi. More than twenty volatile compounds identified in dried ogi and dried maize grains significantly ($p<0.05$) reduced up to 95-99% when compared with fresh wet ogi. The first and second principal components for dried ogi had variances of eigenvalue 32.04 and 1.92, respectively. The study revealed and established the challenges of retaining important volatile compounds responsible for aroma flavour in dried ogi product, most especially with the use of a cabinet dryer.

1. Introduction

The relevance of drying operation in food processes is particularly mostly applied to food preservation, food product development, distribution and transportation (Hawlader *et al.*, 2006; Hadrich *et al.*, 2008). The reduced water activity during drying of food materials helps prevent undesirable reactions, deterioration and spoilage. By this, the Shelf-life of food material is increased, transportation and distribution of food are made easy as a result of volume reduction (Moraga *et al*., 2004; Hawlader *et al.*, 2006; Shi *et al.*, 2008; Bolaji, Oyewo and Adepoju, 2014). However, physical changes, chemical reactions and alteration in some sensory characteristics were reported by some researchers as a possibility during this process (Díaz-Maroto *et al.,* 2003; Zielinska and Michalska 2016; Huang *et al.,* 2016; Bolaji *et al.,* 2017a). The drying temperature, humidity, flow direction and intensity (flow rate) of the drying air, area of the exposed surface of the food particle, composition and structure of the food have been identified as important factors in the drying process (Hawlader *et al.*, 2006; Hadrich *et al.*, 2008). These are capable of determining the efficiency of the process and the quality of the product.

In view of the importance of ogi and the derived food products, an increase in working class mothers and demand for ogi in the urban region of most west Africa countries, large scale production is appearing indispensable. Ogi slurry is not shelf-stable independently without some necessary action taken. Apart from the traditional method of daily decantation of water, refrigeration is often applied. Unfortunately, this requires a constant power supply which is not always available. This makes dried ogi with potential shelf-life stability a needed option. In the past, drying of ogi was attempted and recommended to increase shelf life (Bolaji, Oyewo and Adepoju, 2014; Bolaji, Akindele and Aina, 2014; Bolaji *et al.,* 2015). However, based on the findings of some researchers, the flavour, aroma and taste usually impacted by the presence of volatile compounds may be affected by dehydration process (Zielinska and Michalska 2016; Huang *et al.,* 2016). Most of the drying of ogi slurry in the recent times either for commercial or research purposes has always been cabinet and oven drying methods (Bolaji, Akindele and

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Aina, 2014, Bolaji *et al.,* 2015). The impact of the drying process and method on the volatile compounds believed to be responsible for the acceptable taste, aroma and flavour need to be investigated. The findings will be useful information needed in handling the ogi slurry during the drying process, help to select possible processing conditions that are capable of aiding conservation of these parameters and may help where possible in the development of ogi flavour from principal volatile compounds.

2. Materials and methods

Thirteen kilograms (13 kg) of maize grains were divided into equal parts of 1 kg each after foreign materials were carefully removed and were soaked for 24 hrs, wet milled and sieved with 212 µm sieves. (Bolaji, Adenuga-Ogunji and Abegunde, 2017). These were allowed to ferment for 24 hrs. The wet ogi samples were dried at varying combination of drying temperature (40, 50 and 60°C) and time (8, 10 and 12 hrs) using a central composite rotatable experimental design by design expert software with two factors and five levels (-1.4142, -1, 0, 1 and 1.4142) as shown in Table 1. The drying process was achieved with the aid of locally fabricated cabinet drier. The drying kinetics was evaluated and the volatile compounds of the dried ogi with the use of Gas Chromatography-Mass Spectrometry (GC-MS) were determined.

2.1 Experimental design and modelling for optimization

Response surface modeling (RSM) comprising of central composite design with two factors and five levels were used as shown in Table 1. A second-order polynomial equation was fitted to determine the relationship between dependent variables and independent variables. The model proposed for the response (Yi) was:

$$Yi=b_0+b_1X_1+b_2X_2+b_{11}X_1^2+b_{22}X_2^2+b_{12}X_1X_2 \quad (1)$$

Where X_1 and X_2 are drying temperatures (°C) and drying time (hrs) respectively; b_0 is the value of the fitted response at the centre point of the design; and Yi is the predicted response. The regression coefficient for the linear, quadratic and interaction are represented by b_1–b_2, b_{11}–b_{22} and b_{12}, respectively. The significance of regression coefficients was accessed by p-value at 0.05 significance levels.

Table 1. Coded values of the independent variables

	-α	-1	0	1	+α
X_1(drying temperature)	42.93	40	50	60	57.07
X_2 (drying time)	8.59	10	12	16	11.41

α = 1.4142. X_1, drying temperature; X_2, drying time.

The moisture content determination of wet and dried ogi was in accordance with AOAC (1990). Drying experiments were expressed in dimensionless form as moisture ratios, MR as shown in equation (2):

$$MR = \frac{M-M_e}{M_i-M_e} = \exp(-kt) \quad (2)$$

Where M is the moisture content at any time; M_i is the initial moisture content; and M_e is the equilibrium moisture content (Aghbashlo *et al.,* 2009; Mirzaee *et al*., 2009).

2.2 Determination of volatile compounds

Volatile compounds determination of wet and dried ogi was achieved using GC-MS method. The extraction of the volatile compound was determined by measuring 5 g of each sample, mixed with 20 mL of distilled water, filtered and centrifuged at 10,000 x *g* for 30 mins. All the volatile compounds of wet and dried ogi and dried maize grain were analysed by using the Agilent Technologies 6890 gas chromatography HP chemstation Rev.A0901 (1206). The Headspace extraction of ogi volatile compounds was within 30 mins at a temperature of 60°C followed by GC separation elaborated with column oven initial temperature at 35°C at the rate of 5°C/min to reach 300°C. Identification of the compounds was by GC retention time against standards.

3. Results and discussion

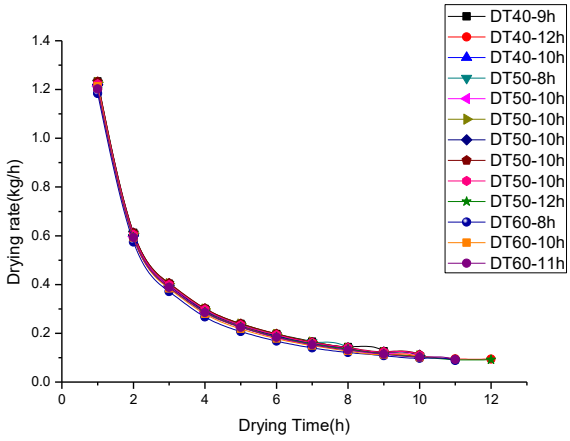

Figure 1. Drying rate of ogi slurry at varying drying condition

The rate of moisture removal from wet ogi with initial average moisture content of 48.31% during drying process of ogi was affected by the surface area, pore spaces and thermal properties of the ogi slurry as reported by some researchers (Moraga *et al.*, 2004; Hawlader *et al.*, 2006; Shi *et al.*, 2008; Bolaji, Akindele and Aina, 2014; Bolaji *et al.,* 2015). As shown in Figure 1 the drying rate continuously decreased with decrease and increase in moisture content and drying time, respectively. This was consistent with the findings of

some researchers (Doymaz, 2005; Bolaji, Akindele and Aina, 2014). According to Zielinska and Michalska (2016), the drying operation may have caused some chemical alterations in ogi during the drying operation.

3.1 Estimation of effective moisture diffusivities

The effective diffusivities of the drying process of ogi were estimated by the method reported for drying characteristics of biological products in falling rate period using Fick's diffusion equation (Perea-Flores *et al.,* 2012) as shown in equation (3):

$$MR = \frac{8}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{n^2}\exp\left[-n^2\frac{\pi^2 D_{eff}t}{r^2}\right] \quad (3)$$

Where MR is the moisture ratio; D_{eff} is the effective moisture diffusivity (m^2/s); r is the equivalent radius (m) and t is the time (s). A logarithmic form is as shown in Equation (4). The effective moisture diffusivity was calculated from the slope of a straight line obtained from the plot of ln(MR) and drying time (Perea-Flores *et al.,* 2012).

$$\ln MR = \ln\frac{8}{\pi^2} - \left(\frac{\pi}{r}\right)^2 D_{eff}\,t \quad (4)$$

The effective moisture diffusivity was estimated by using the method of slopes. From Equation (3), a plot of ln(MR) versus drying time gave a straight line and the slope was determined using equation (5):

$$Slope = \frac{\pi^2 D_{eff}}{r^2} \quad (5)$$

Table 2. Effective moisture diffusivity of ogi at experimental drying condition

Drying conditions	Final moisture content (%)	Effective diffusivity
40°C - 9 hrs	30.96	-64
40°C - 12 hrs	8.98	-1.3
40°C - 10 hrs	20.52	-1.3
50°C - 8 hrs	26.96	-99
50°C - 10 hrs	7.86	-2.2
50°C - 10 hrs	18.6	-1.1
50°C - 10 hrs	17.72	-1.1
50°C - 10 hrs	25.52	-84
50°C - 10 hrs	20.54	-91
50°C - 12 hrs	7.4	-1.4
60°C - 8 hrs	7.44	-8
60°C - 10 hrs	3.86	-3.3
60°C - 11 hrs	5.68	-3.28

The computed effective moisture diffusivity (D_{eff}) of ogi is as shown in Table 2. These values were comparable with values obtained from some researchers (Babalis and Belessiotis, 2004; Aghbashlo *et al.,* 2009; Mirzaee *et al*., 2009). Research reports indicated possible factors like drying conditions, experimental procedures, data treatment methods, temperature, product properties, compositions, physiological state and heterogeneity of structure may affect effective moisture diffusivity during dehydration (Babalis and Belessiotis, 2004; Aghbashlo *et al*., 2009; Mirzaee *et al*., 2009). The fineness of ogi and its closely packed structure may have affected the entire drying kinetics and effective moisture diffusivity as well.

3.2 Volatile compound

The average retention time for respective detected volatile compounds of ogi produced from maize grains at designed experimental condition (drying temperature and drying time are as shown in Table 3. The volatile compounds of ogi before drying and dried maize grains were as well included. A total of thirty-five volatile compounds were detected in wet ogi, dried ogi and dried maize grains. The retention time ranged from 9.67 to 26.96 mins. There was no significant difference ($p<0.05$) in the retention time of the same volatile compounds across the experimental condition for wet and dried ogi as well as dried maize grain. The acetaldehyde found in fresh ogi, dried ogi and dried maize grain recorded retention time ranging from 9.69-9.86 mins while the last detected volatile compound, ethyl caprate retention time for wet ogi, dried ogi and dried maize grain range between 25.05-26.67 mins. The number of these volatile compounds were lower than values reported for fermented dough and yoghurt, respectively (Annan *et al.,* 2003; Cheng, 2010).

The acetaldehyde, ethyl butyrate, 2-methyl butanol, ethanol, hexanal, isoamyl acetate, 2,3-butanediol, propanoic acid, ethyl caproate, 2,3-butanedione, 3-methyl butanol, 2-methyl propanol, heptanol, acetic acid, propyl acetate and propanol reduced by 99.99% while nonanal reduced by 99.88%, hexadecenoic acid by 97.93%, ethyl acetate by 97.25%, 2-methyl-naphthalene by 95.02%, E-2-nonenal by 96.62%, 2-methoxyphenol by 60.43%, ethyl dec-9-enoate by 87.38% and ethyl lactate by 10.36%. This was consistent with the reports from some researchers (Baranauskiene *et al.,* 2006; Calin -Sanchez *et al.,* 2011; Huang *et al.,* 2016). The losses recorded in this work were higher than values reported by these researchers. However, isobutyl acetate, i-methylnaphthalene, ethyl isovalerate, ethyl caprylate, ethyl valerate 2-Nonenal and 2-methoxy-4-vinylphenol increased in dried ogi and dried maize grains when compared with these volatile compounds in wet ogi. The concentration of respective volatile compounds in dried ogi compared with respective concentration in wet ogi is as shown in Figure 2. These losses of the volatile compounds may have contributed to the observation of usual deviation in aroma and flavour of dried ogi from fresh ogi slurry in earlier research works (Bolaji, Oyewo and Adepoju, 2014, Bolaji *et al.,* 2015). The contor response surface model plot in Figure 3 revealed that all

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Table 3. Detected volatile compounds and mean retention time (min.) of ogi produced from maize grains at 24th soaking period of maize grains and designed drying condition

	Wet-fresh ogi	Dried maize	40°C - 9 hrs	40°C - 12 hrs	40°C - 10 hrs	50°C - 12 hrs	50°C - 8 hrs	50°C - 10 hrs	50°C - 10 hrs	50°C - 10 hrs	50°C - 10 hrs	50°C - 10 hrs	60°C - 8 hrs	60°C - 11 hrs	60°C - 10 hrs
Acetaldehyde	9.71	9.72	9.87	9.696	9.692	9.74	9.75	9.74	9.74	9.73	9.75	9.86	9.73	9.86	9.69
Ethanol	10.5	10.64	10.38	10.63	10.64	10.64	10.36	10.64	10.65	10.64	10.65	10.59	10.64	10.5	10.63
Acetic acid	11.35	11.35	11.45	11.35	11.35	11.35	11.36	11.35	11.35	11.35	11.35	11.29	11.35	11.58	11.35
Propanol	12.82	12.82	12.94	12.82	12.82	12.82	12.83	12.81	12.82	12.82	12.82	12.83	12.82	13.03	12.82
2-methyl propanal	13.54	13.42	13.43	13.42	13.42	13.42	13.42	13.41	13.42	13.41	13.42	13.65	13.42	13.4	13.42
2-methyl propanol	14.15	14.15	14.14	14.15	14.15	14.15	14.15	14.14	13.97	14.15	14.15	14.9	14.15	14.15	14.15
2,3-butanedione	14.33	14.43	14.77	14.43	14.43	14.7	14.43	14.43	14.15	14.43	14.7	14.4	14.43	14.7	14.43
3-methyl butanal	15.03	14.7	15.027	15.04	14.7	15.05	14.06	14.91	14.91	15.05	14.91	14.91	14.7	15.03	14.7
3-methyl butanol	15.39	15.5	15.624	15.5	15.5	15.5	15.51	15.5	15.51	15.51	15.5	15.37	15.5	15.62	15.5
2-methyl butanol	15.62	15.89	15.89	15.89	15.89	15.89	15.89	15.89	15.89	15.89	15.85	15.67	15.89	15.8	15.89
Hexanal	16.05	16.04	16.04	16.04	16.04	16.04	16.04	16.04	16.04	16.04	16.04	15.98	16.04	16.04	16.04
Ethyl acetate	16.47	16.45	16.5	16.56	16.45	16.48	16.46	16.48	16.49	16.48	16.48	16.65	16.37	16.56	16.45
2, 3-butanediol	16.78	16.78	16.788	16.78	16.78	16.78	17.78	16.67	16.78	16.78	16.79	17.01	16.78	16.79	16.78
Heptanol	17.36	17.09	17.09	17.09	17.09	17.09	17.09	17.09	17.09	17.09	17.09	17.24	17.09	17.09	17.09
Propyl acetate	17.54	17.54	17.55	17.47	17.54	17.54	17.55	17.54	17.54	17.55	17.6	17.47	17.55	17.55	17.55
Propanoic acid	17.67	17.77	17.78	17.77	17.77	17.77	17.77	17.77	17.77	17.77	17.77	17.81	17.77	17.77	17.77
Hexadecanoic acid	18.05	18.05	18.14	18.05	18.05	18.05	18.05	18.06	18.05	18.05	18.05	17.98	18.05	18.05	18.05
Isobutyl acetate	18.49	18.59	18.36	18.35	18.59	18.59	18.59	18.58	18.58	18.58	18.35	18.3	18.58	18.59	18.59
Ethyl butyrate	18.93	18.93	18.59	18.59	18.92	18.93	18.92	18.92	18.93	18.92	18.59	18.55	18.92	18.93	18.92
Nonanal	19.09	19.1	19.11	19.1	19.1	19.1	18.1	19.09	19.09	19.09	19.1	18.99	19.09	19.1	19.1
2-methylnaphthalene	19.53	19.52	19.53	19.52	19.52	19.52	19.52	19.51	19.52	19.52	19.52	19.34	19.52	19.52	19.52
Ethyl lactate	19.88	19.69	19.68	19.88	19.69	19.68	19.88	19.88	19.67	19.67	19.68	20	19.69	19.88	19.6
2-methoxyphenol	20.19	20.11	20.12	20.11	20.11	20.11	20.11	20.11	20.1	20.11	20.11	20.33	20.11	20.11	20.11
(E)-2-nonenal	20.53	20.46	20.54	20.47	20.46	20.47	20.47	20.47	20.47	20.47	20.47	20.69	20.46	20.48	20.46

Table 3. Detected volatile compounds and mean retention time (min.) of ogi produced from maize grains at 24th soaking period of maize grains and designed drying condition (Cont.)

	Wet-fresh ogi	Dried maize	40°C - 9 hrs	40°C - 12 hrs	40°C - 10 hrs	50°C - 12 hrs	50°C - 8 hrs	50°C - 10 hrs	50°C - 10 hrs	50°C - 10 hrs	50°C - 10 hrs	50°C - 10 hrs	60°C - 12 hrs	60°C - 11 hrs	60°C - 10 hrs
1-methylnaphthalene	20.79	20.79	20.799	20.79	20.79	20.79	20.79	20.79	20.79	20.79	20.79	20.95	20.79	20.79	20.79
Ethyl isovalerate	20.98	20.99	21.14	21.01	21.01	20.99	20.99	20.99	20.99	20.99	21.12	21.12	20.99	20.99	21.9
Isoamyl acetate	21.44	21.66	21.323	21.44	21.32	21.45	21.45	21.44	21.31	21.44	21.82	21.47	21.32	21.66	21.32
2-methylbutyl acetate	22.06	22.08	22.07	22.08	22.08	22.08	22.08	22.07	22.06	22.07	22.07	22.99	22.07	22.08	22.078
Ethyl valerate	22.56	22.6	22.61	22.6	22.59	22.59	22.6	22.59	22.6	22.59	22.59	23.35	22.6	22.61	22.59
2-nonanone	22.99	23.12	23.12	23	23.12	23.12	23.12	23.11	23.11	23.11	23.1	23.08	23.11	23.12	23.12
Ethyl caproate	23.62	23.97	23.48	23.47	23.97	23.47	23.47	23.47	23.47	23.47	23.82	25.49	23.47	23.48	23.47
2-methyl-4-vinylphenol	25.35	24.99	25.69	24.99	24.99	25.09	25.09	24.09	25.69	25.69	24.08	25.16	25.99	25	25.69
Ethyl caprylate	26.5	26.05	26.29	25.97	25.97	26.06	26.06	26.05	26.28	26.28	26.06	25.94	26.97	26.05	26.29
Ethyl Dec-9-enoate	26.95	27.34	27.058	26.3	26.29	26.29	26.29	27.28	26.99	27.05	27.28	26.3	26.32	26.35	27.06
Ethyl caprate	27.43	27.29	27.44	27.29	27.29	27.43	27.43	27.43	27.43	27.42	27.43	27.4	27.29	27.46	27.29

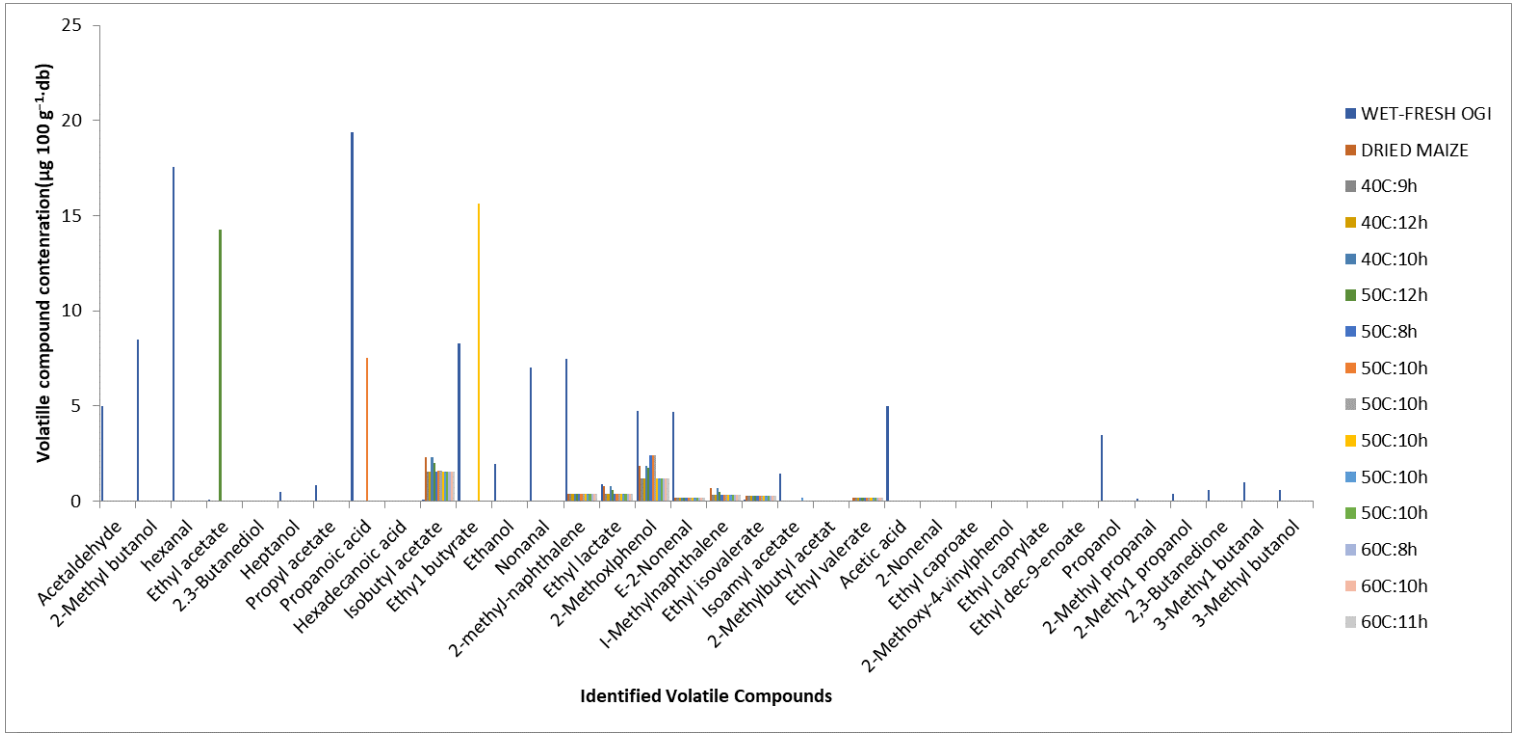

Figure 2. Concentration of volatile compounds of wet ogi compared with dried ogi at varying drying conditions

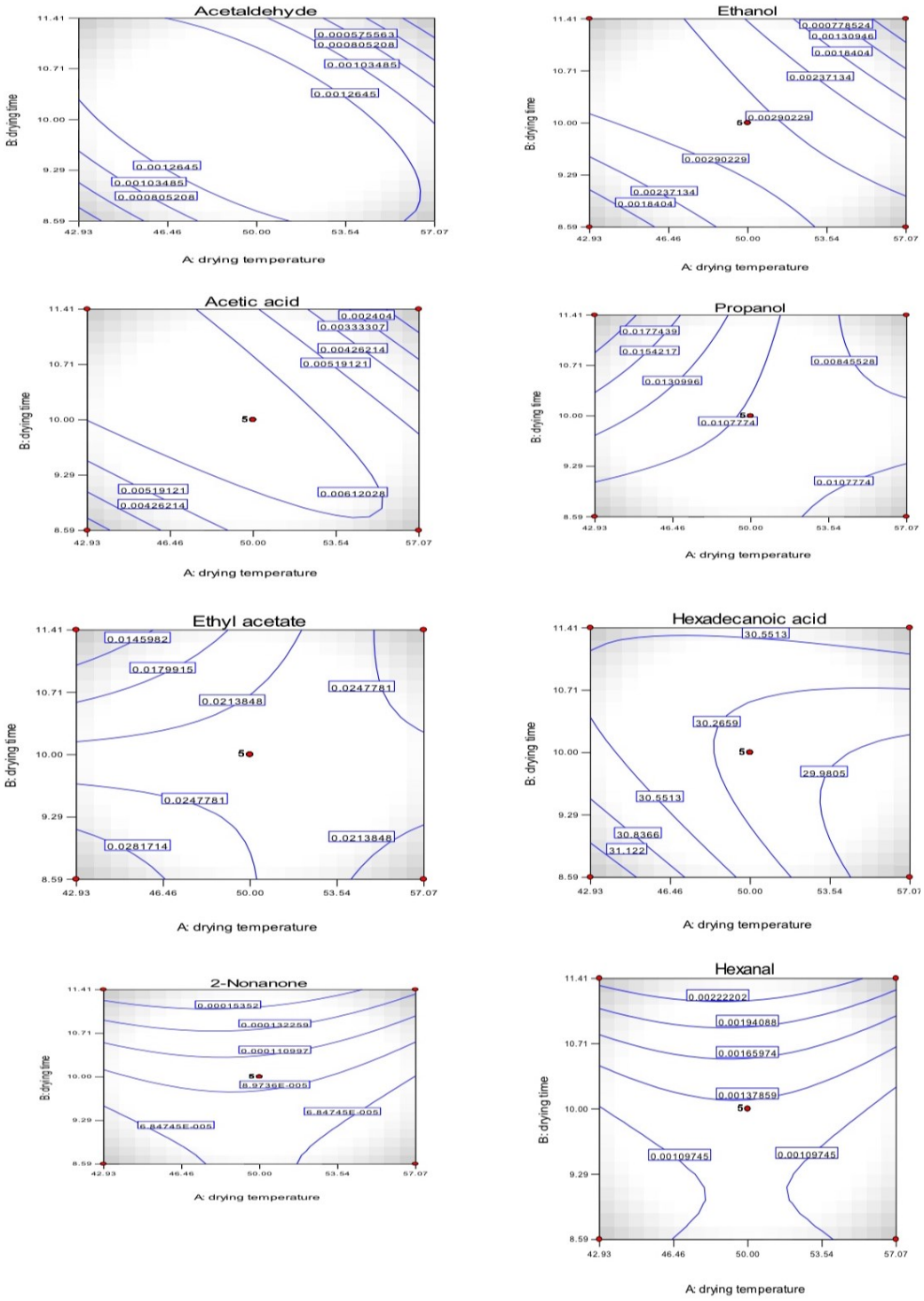

Figure 3. Contour plot of some volatile compounds' reduction at varying drying condition of temperature and time when compared with fresh ogi product

the volatile compounds decreased with increased drying temperature and time. According to Azime *et al.* (2018), the drying techniques and condition in this research work may have encouraged the losses of volatile compounds. According to Huang *et al.* (2016), the lower drying temperature may be advantageous. The results obtained for some volatile compounds showed that they were thermally unstable. Unlike the report by Toontom *et al.* (2016), there were no outright disappearances of some volatile compounds detected during and after drying in this work, but the gross reduction was evidently recorded. The increase in acetic acid after drying for Thai chilli's operation reported by Toontom *et al.* (2016) was completely contrary to the observation in this study.

The first principal component for dried ogi had a variance of eigenvalue 32.04% and account for 91.5% while the second principal component had a variance of 1.92 accounted for 5.70%. The third and fourth principal components had variances 0.975 and 0.011, (2.80 and 0.00%), respectively. The principal components showed that few volatile compounds were relevant on the flavour and aroma of the dried ogi since some with higher per cent concentration detected in freshly produced ogi had grossly reduced by drying operation employed in this work.

4. Conclusion

As expected, the drying rate of the operation decreased with increased drying time. About fourteen out of the thirty-five volatile compounds detected in the dried ogi and dried maize grains significantly ($p<0.05$) reduced up to 99.99% when compared with wet ogi. While nonanal, hexadecanoic acid, ethyl acetate, 2-methyl-naphthalene, E-2-nonenal, 2-methoxlphenol, ethyl dec-9-enoate and ethyl lactate reduced by 99.88%, 97.93%, 97.25%, 95.02%, 96.62%, 60.43%, 87.38% and 10.36% respectively. The concentration of respective volatile compounds in dried ogi compared with respective concentration in wet ogi suggested the loss of aroma and flavour. This work established that some of the volatile compounds detected in this work were thermally unstable. In order to retain the volatile compounds in ogi during drying operation, there may be a need to develop an improved or employ drying process capable of reducing the losses of relevant volatile compounds experienced in this work.

Conflict of Interest

The authors declare no conflict of interest.

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