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Effect of process variables on the sterilization value, F_o of glass canned indigenous soups

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ABSTRACT: The study examined how soup variety, retorting temperature, and time affect the F_o, which measures heat penetration and sterilization during canning. Two renowned South-Eastern Nigerian delicacies, Egusi and Ogbono soups, were cooked, bottled, and sterilized in glass containers. The purpose of this study was to determine how the thermal processing conditions and soup variety affect the degree of sterilization of soups made for convenience and longer shelf life without cold storage. The soups were made using conventional ingredients and procedures, then poured into glass jars and sterilized at temperatures ranging from 110°C to 121°C for 60 to 90 min. The heat uptake by the soups at the glass cans' presumed slowest heating zones was measured using thermocouples positioned in the centre of the can. The soups were sterilized in batches based on the temperature and time combinations. Periodic temperature and heat uptake measurements were utilized to calculate the process's F_o. The F_o ranged from 1.0494 to 40.1739 min, with egusi soup demonstrating faster heat uptake and greater F_o in most sterilization regimes. To ensure botulinum cook in the soups, heat processing at a least of 115 °C was required. Only the sterilization temperature significantly ($p<0.05$) affected the degree of sterilization, F_o. The linear model significantly described the F_o data, with acceptable R^2 (0.8579), adjR^2 (0.8275), predR^2 (0.7653) and adequate precision (12.7227). The F_o model could be a reliable guide in setting retorting condition for canned indigenous soups.

Keywords: Soup, Glass canning, Thermal processing, Sterilization, Heat penetration, F_o
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INTRODUCTION

In Africa, the most common way of consuming leafy vegetables is by using them in preparing soups. The utilization of leafy vegetable is part of Africa's cultural heritage, as they play important roles in the customs, traditions and food culture of the African household (Sanusi and Olurin, 2012). Soups occupy a significant component of diets in Nigerian homes (Bamidele *et al.*, 2017). There is a growing demand for ready-to-eat and easy-to-use convenience products due to social and cultural changes in recent years (Pagarkar *et al.*, 2011), where traditional consumers also demand their traditional cuisines in convenient forms. In addition to safety, consumers expect extended shelf-life and high convenience in preparation and use (Taoukis and Giannakourou, 2004). Thermal processing by canning and sterilization remains a famous effective method of

prolonging the shelf life of foods, making them convenient, affordable, easy to transport, and making the foods available (Tucker, 2016; Simpson *et al*., 2020). In thermal processing, the study of heat penetration into foods is of great importance. Among the factors that influence the rate of heat penetration are the container properties, retort temperature and nature of the product (Al-Baali and Farid, 2006). The impact on the sterilization then follows how long the food is subjected to that retort temperature. One important quantitative parameter in the thermal processing is the thermal death time (F_0), or F value. It is defined as the time required to destroy a given percentage of microorganisms of given Z value at a given temperature. How the product being sterilized in the given container takes up heat shows in the F value. This parameter states the reduction in the population of microbial pathogens required to establish product safety. The F value represents the total time-temperature combination received by a food and is quoted with suffixes indicating the retort temperature and the Z value of the target microorganism (Al-Baali and Farid, 2006). A reference F value, F_{ref} is used to describe processes that operate at 121 °C, which are based on a microorganism with a Z value of 10 °C. The F value is defined by the relationship between F_{ref} and the change in the concentration of microorganism in a containerized food, as a result of heating or cooling (David *et al*., 1996).

Sterilizations in retort processing are characterized by four distinct stages. Firstly, the food has to be heated to a temperature of 110 °C – 125 °C to ensure sterilization. Secondly, the product needs a few minutes to equilibrate, as the surface would be hotter than the central portion of the container creating a temperature gradient. The equilibration stage allows a drop in the temperature gradient. Thirdly, the product has to be held at this temperature for certain duration to ensure a predetermined sterilization value designated by F_o value. Finally, the product has to be cooled mainly to seize further heat treatment and avoid over cooking (Harlfinger, 1992). As *Clostridium botulinum* spores can proliferate in low-acid (pH>4.6) products during storage, the food, therefore, must be thermally treated to the equivalent of at least 121.1 °C for 3 min (an F_0 value of 3) to achieve a 12-decimal reduction of the microorganism. The processing conditions should be applied to the slowest-heating point referred to as “cold point.” This makes possible the assumption that, success in sterilizing the slowest heating part by exposing it to the required time–temperature profile, assures that the rest of the product would have been sterilized (Ramesh, 2007).

Practically, the sterilization would lead to impacts on the product quality and nutrients. These effects are mostly deteriorative (Ramesh, 1995), while destroying microorganisms to render the product safe. The heat treatment used, therefore, depends on knowing the time-temperature combination required to inactivate the most heat-resistant pathogens and spoilage organisms in the particular food (Al-Baali and Farid, 2006; Toledo, 2007; Garg, 2019) without much deleterious impacts on the product quality and nutrients. As glass is a poor conductor of heat, it is therefore pertinent to study the heat uptake by the glass-canned soups and to establish which variables significantly influence the extent of sterilization of the soups, evident in the F values (F_0).

MATERIALS AND METHODS

Raw and packaging materials acquisition

Ingredients for the soups were purchased from a local market in Umuahia, Abia State, Nigeria. For the packaging material for the canning, empty old and used glass jars were obtained from a local market where old containers are vended in Aba, Nigeria. The collected glass jars and metal caps were washed with detergent solution and thoroughly rinsed with potable water. The washed jars and caps were dried in a hot air oven at 160 °C for one hour, after which the heat was turned off and the oven temperature was allowed to drop to 80°C before being opened and the containers and caps removed and kept for use.

Experimental design and response surface modelling of the F_0 of the soups

The experiment was laid on a three-factor (2x3x3) factorial setting, with soup (A), sterilization temperature (B) and sterilization time (C) being the factors X_1, X_2 and X_3, respectively. Data for the response variable were subjected to multiple linear regressions analysis using Design Expert® 11.0 software (StatEase, Minneapolis). The data were fitted to different orders of polynomial model. Model (mathematical equation) was developed for the response. The acceptability of the models was based on the model adequacy checks, significance of the model, high coefficient of determination (R^2), adjusted R^2, predicted R^2, and acceptable adequate precision. The highest order polynomial with acceptable model adequacy properties was selected. The regression model, as reported by Idowu (2014), followed Equation 1.

$$Y=\beta_0+\beta_1X_1+\beta_2X_2+\beta_3X_3+\beta_{11}X_1^2+\beta_{22}X_2^2+\beta_{33}X_3^2+\beta_{12}X_1X_2+\beta_{13}X_1X_3+\beta_{23}X_2X_3+\varepsilon \quad (1)$$

Where Y is the dependent variable (F_0); X_1 – X_3 are coded independent variables, and X_1 = Soup variety, X_2 = Sterilization temperature (°C) and X_3 = Sterilization time (min); β_0 is the equation regression coefficient for

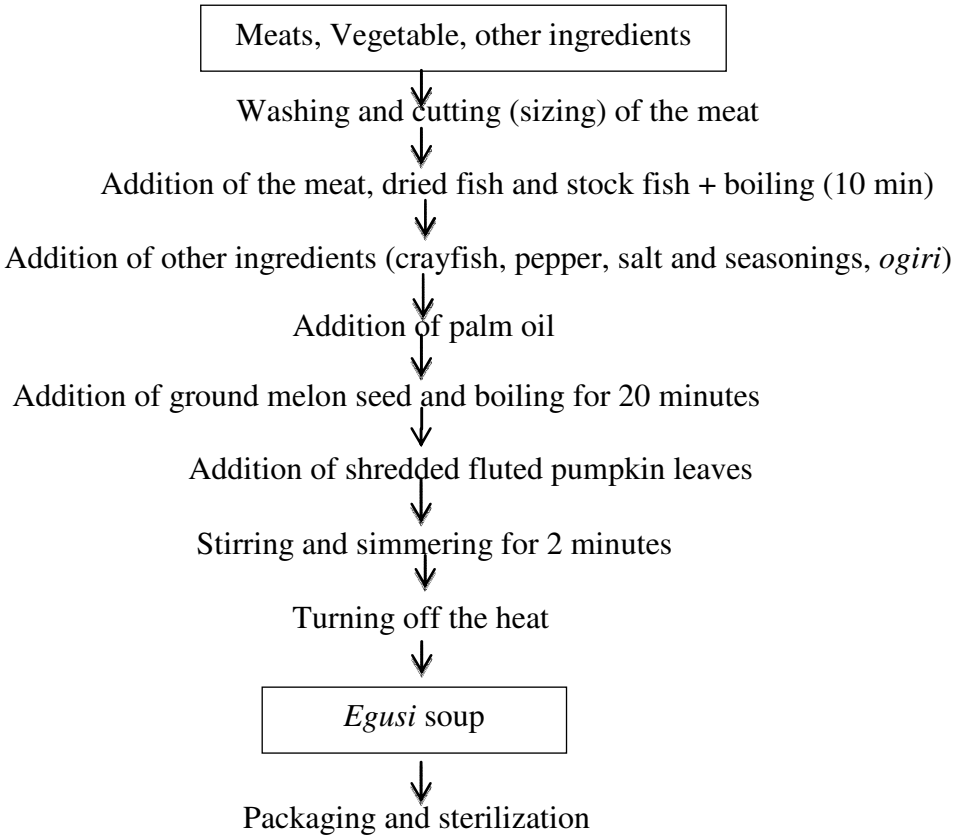

Figure 1: Flow chart for the preparation of canned Egusi soup (Omah *et al*., 2015 modified).

intercept; $\beta_1 - \beta_3$ are the equation regression coefficients for linear effects; β_{11}-β_{33} are the equation regression coefficients for quadratic effects; $\beta_{12} - \beta_{23}$ are the equation regression coefficients for interaction effects; ε is the error term.

Soup recipes/formulation and preparation

The ingredients were according to the traditional recipes, as often used for Egusi and Ogbono soups by the locals. Fresh fluted pumpkin (*Telfaria occidentalis*) leaves were used as the vegetable in the soups. Egusi soup was prepared according to the recipe and method described by Omah *et al*. (2015) with a few modifications. The method of preparation was as presented in (Figure 1). A modified recipe for ogbono soup by Federal Institute of Industrial Research, Oshodi (FIIRO) reported by Raji *et al*. (2016) was adopted. The ogbono soup was prepared according to the method described by Faleti (1999), with slight modification, as shown in (Figure 2).

Sterilization, heat uptake and F_0

The sterilization of the soups was done using the method described by Toledo (2007), with modifications. Here, the soups were manually filled into the glass jars and securely capped. They were loaded into the sterilization chamber of the retort. According to the sterilization temperature-time combinations in the experimental design, the thermal processing went in nine (9) batches for the eighteen (18) experimental runs, with each batch having both soup varieties. Heat penetration monitoring cans were inserted in each batch for each soup variety. The retort was securely closed, turned (ON) and the sterilization temperature and time were set using the operations buttons on the equipment.

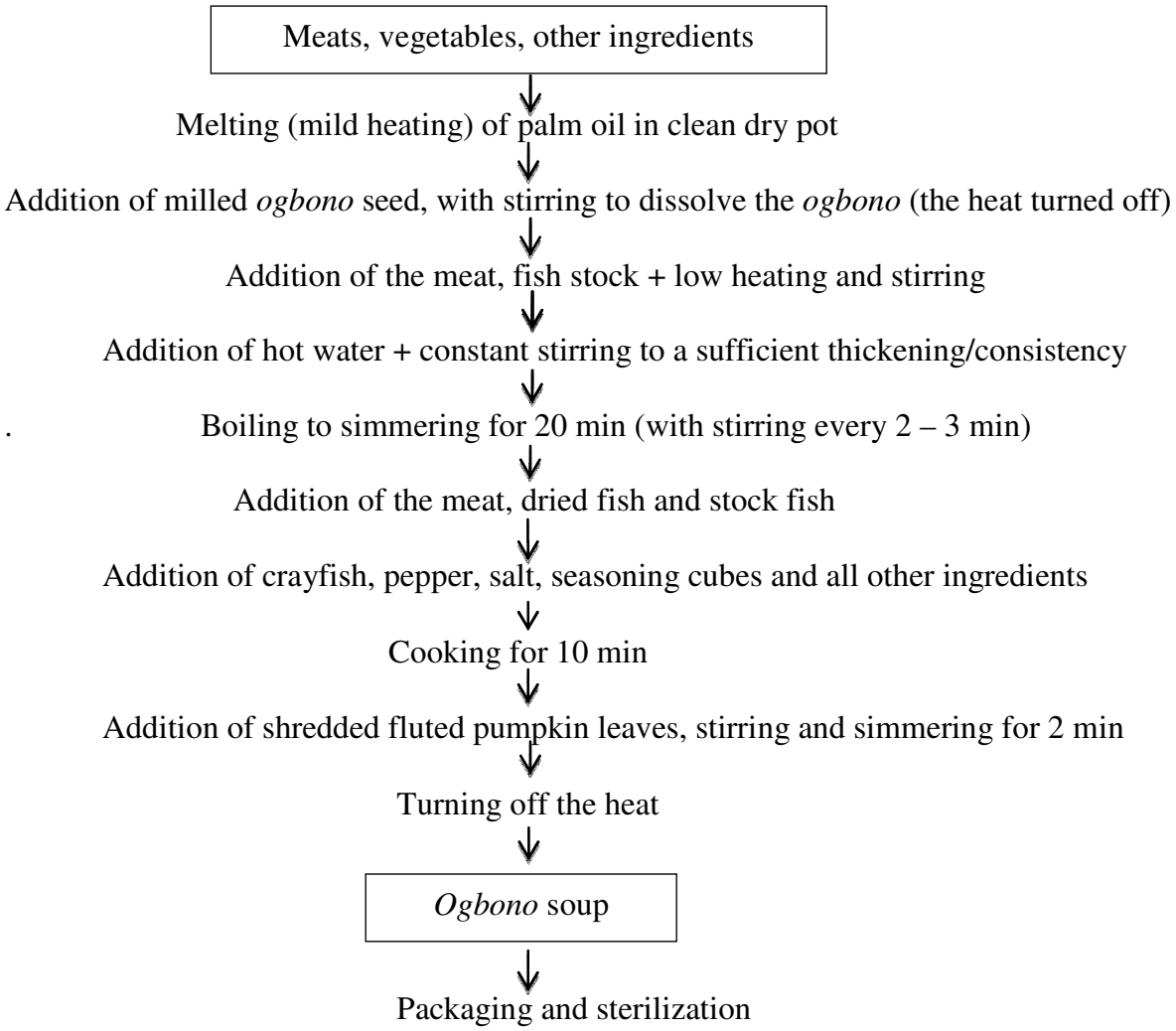

Figure 2: Flow chart for the preparation of canned Ogbono soup (Faleti, 1999, modified).

When equipment was exhausted as automatically set when the retort temperature reached 100°C. This was to remove excess air in the retort. After exhausting, the temperature started rising to that set for the sterilization batch. This counted for the come-up time (CUT). When the set temperature was attained, the sterilization started, and lasted for the set time. When the sterilization was completed according to the set time, the (STOP) button was pressed and the process stopped. The pressure vents were opened and the pressure dropped. The equipment lid was opened and the samples brought out and air-cooled.

The variables, Lethality and F value (F_0), were estimated using the method described by Toledo (2007). For each batch of sterilization of the soups, one of the glass cans was labelled "Heat penetration." A thermocouple was inserted at the centre of the heat penetration can and placed in the autoclave during the sterilization. Temperature at the centre of the can, showing heat uptake by the product, was taken with the thermocouple at intervals of two (2) minutes. The data were inputted in a Microsoft Excel sheet and computation algorithm was set for the calculation of the process lethality at each time, t, from the temperature recorded for that time, assuming a standard z-value of 10. The total (sum of the) process lethality, corresponding to the area under the curve of a Time-Temperature graph, gave the F_0 of the process.

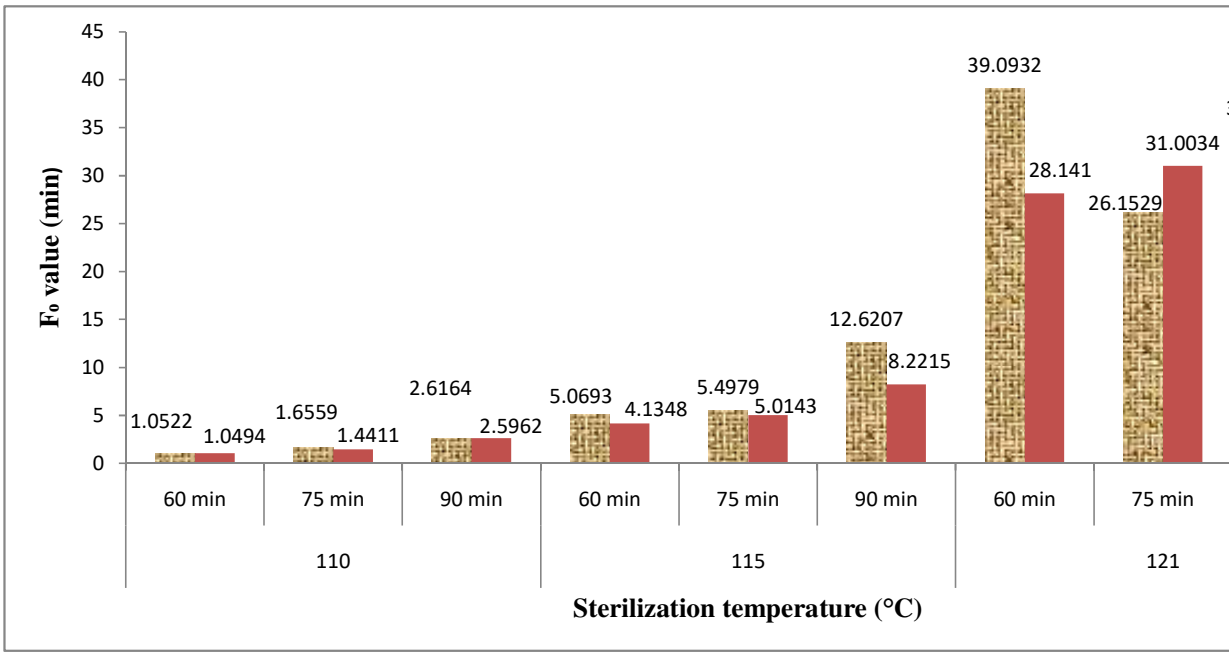

Figure 3: F_0 values of glass canned indigenous soups at different sterilization regimes.

Mathematically, the lethality and F_0 were obtained as:

$$\text{Lethality, L at time t} = 10^{(Ti-121)/z} \quad (2)$$

and;

$$\text{F value, } F_0 = \Sigma L \quad (3)$$

Where T_i = Temperature at time t; z = z-value of *Clostridium botulinum* spore, assumed to be 10.

RESULTS AND DISCUSSION

Figure 3 shows the sterilization values (F_0) derived from different temperature-time regimes. The heat penetration provides an indication of the potential thermobacteriological influence of sterilizing indigenous soups. It also indicates the likelihood of nutritional degradation and lipid oxidation. The heat penetration data from various processing regimes revealed that, despite varying initial temperatures, the rate of heat uptake and temperature at the coldest spots of the two soups remained nearly equal at ≥30 minutes into sterilization. However, the point lethality of the beginning temperature would continue to influence the subsequent F-values, as high initial temperatures contributed to a high ultimate F-value.

Generally, for sterilization at 110 – 115 °C, egusi soups consistently showed slightly higher process lethality (F-values, F_0) than ogbono soup samples. The heat uptake by the egusi and ogbono soups sterilized at 110 °C gave respective F_0 of 1.0522 and 1.0494 min after 60 minutes sterilization, 1.6559 and 1.4411 min after 75 minutes sterilization, and 2.6164 and 2.5962 min after 90 minutes sterilization. The sterilization regimes at 115 °C for 60, 75 and 90 minutes had respective F_0 of 5.0693 and 4.1348 min, 5.4979 and 5.0143 min, and 12.6207 and 8.2215 min for egusi and ogbono soups. For sterilization at 121 °C, the F_0 values of egusi ranged from 26.1529 to 39.0932 min, while the F_0 values in the ogbono soup ranged between 28.141 and 40.1739 min. However, the kind of soup was not significant ($p>0.05$) in effect (Table 1), signifying that the F_0 values were not necessarily achieved because of the kind of soup involved, but rather because of other significant factors. Although the difference in the initial temperature values may have contributed to the final F_0 of egusi soup being commonly but insignificantly higher than ogbono, the higher viscosity (corresponding with thickness) of the soups may be highly implicated in the slower heat penetration of the ogbono soup, hence the apparent lower F_0 of the ogbono soups. An observation of significantly ($p<0.05$) higher viscosity of the ogbono soup, as was observed in the study, lent credence to this. Gaze (2005) and Al-Baali and Farid (2006) showed that lower thickness and the usual corresponding lower viscosity would give higher and faster heat uptake, and liquid or particulate foods would heat faster than solid foods.

Fasogbon *et al*. (2022) reported that ogbono soup

Table 1: Analysis of variance (ANOVA) of linear model for the F_0 Value of glass canned indigenous soups.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	3130.71	3	1043.57	28.18	< 0.0001	Significant
A-Soup	2.46	1	2.46	0.0664	0.8003	
B-Sterilization temperature	3086.58	1	3086.58	83.35	< 0.0001	
C-Sterilization time	41.67	1	41.67	1.13	0.3068	
Residual	518.46	14	37.03			
Corrected Total	3649.17	17				

F_0 = +14.39-0.3697A+16.02B+1.86C … Eqn 4

R^2 0.8579; Adjusted R^2 0.8275; Predicted R^2 0.7653 Adeq precision 12.7227
PRESS 856.30; Mean 13.90; Std. Dev. 6.09; C.V. % 43.78 ; -2 Log Likelihood 111.57; BIC 123.13; AICc 122.65. The Predicted R^2 of 0.7653 is in reasonable agreement with the Adjusted R^2 of 0.8275; i.e., the difference is less than 0.2. Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 12.7227 indicates an adequate signal. This model can be used to navigate the design space.

sterilized at 110 °C, 116 °C and 121 °C had F-values of 31.63 – 61.38 min within a maximum processing time of 31 min. The treatment delivered, respectively, a 54%, 71%, and 75% log-reduction of the surviving *Bacillus stearothermophilus* spores after thermal processing of the canned dika kernel (ogbono) soup. Various F_0 reported for some commercial canning processes for meat and vegetable laden foods ranged from 2 – 15 minutes, with values varying with can size, meat and vegetable types. Typical F_0 values reported by Fellows (2000) for closely related products are 3 – 6 min for vegetables in brine, 4 – 5 min for cream soups and 12 – 15 min for meat in gravy. For others, vegetables such as asparagus had F_0 of 2 – 4 min. Mackerel in brine had values 2.9 – 3.6 min, while sausage, Vienna in brine and meat loaf had F_0 of 5 – 6 min (Toledo, 2007). Richardson (2004) reported that the F_0 for ready-to-eat foods is in the range 5.0 – 30.0 min. Values obtained in this study are close to the range. Although a minimum F_o value of 3.00 has been considered as the process target, based on the heat resistance of *C. botulinum* spores, to be capable of destroying at least 12 decimal reductions of this microorganism, the size of the batch of packages and the retort process capacity, which were not considered in that postulation, could upset the expected success of that sterilization (Gumerato and Schmidt, 2009).

The F_0 values show the total lethality of the temperature-time combinations of the sterilization process. This thermobacteriological parameter, for each sterilization regime, is equivalent in minutes to the lethal effect of heating the soups at a designated reference temperature (often, and in this case, 121 °C) and at assumed *z* value of 10, assuming instantaneous heating to this temperature and instantaneous cooling from this temperature (Heinz and Hautzinger, 2007). From the F_0 obtained in this study, sterilizing the soups at 110 °C for 60 – 75 min would not give a lethality that suffices for minimum botulinum cook for canned low acid foods. The most-resistant spores of botulinum so far studied (belonging to the proteolytic, halotolerant and mesophilic group I of *C. botulinum* requiring minimum growth temperature ~10 °C) are characterized by a D_{121} of 0.21, requiring a minimum F_0 of 2.52 min in the 12D concept of sterilization (Dalgaard, 2006). Therefore, at 110 °C, the 90 minutes sterilization would be much required to get the soups to minimum commercial sterility in canning standards, based on results obtained in this study.

The sterilization regime of 121 °C for 60 minutes followed the same trend of higher F_0 in egusi (39.0932 min) than ogbono soup (28.1410 min). However, the ogbono soup sterilized at 121 °C for 75 and 90 minutes had respective higher F_0 (31.0034 and 40.1739 min) than their egusi soup counterparts (26.1529 and 34.6719 min) at corresponding equal processing time. The higher initial temperature of the ogbono soup can be implicated in this. The place of the temperature of the soups at commencement of processing (i.e initial temperature) in the final F_0 of the soups was more clearly demonstrated in the 121 °C/60 min sterilization, which had higher F_0 values than those sterilized for even longer period of 75 minutes, for both soups. The initial temperature of the food greatly influences a given lethality and the heating (time) required to administer the lethality: the higher the initial temperature, the shorter the time required to bring about the lethality. Where a fixed processing time is considered relative to different initial temperatures of the food being sterilized, higher initial temperature gives higher final lethality.

The preservative effect of heat processing is due to the denaturation of proteins, which destroys enzyme activity and enzyme-controlled metabolism in microorganisms. The rate of destruction is a first-order reaction; that is when food is heated to a temperature that is high enough to destroy contaminating microorganisms, the same percentage die in a given time interval regardless of the numbers present initially (Fellows, 2000).

Overall, the F_0, which followed the general trend 110 °C < 115 °C < 121 °C for corresponding equal processing

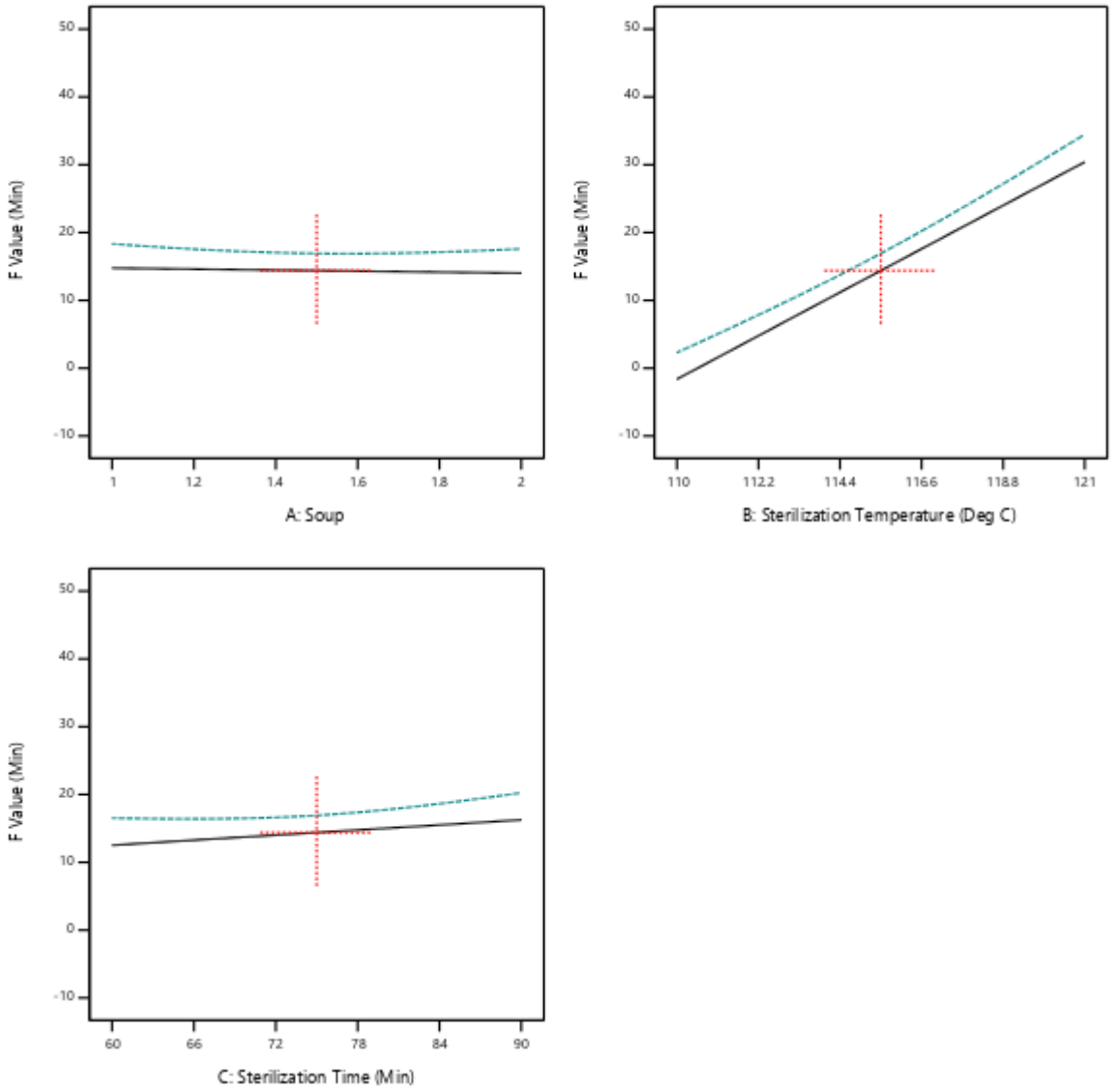

Figure 4: Single factor plots of effects of soup type and sterilization temperature and time on the F_0 of glass canned indigenous soups.

times, were in line with the principle that heat uptake by the sterilized food increases with the temperature of the retort. The heat penetration is not only higher, but also faster, because higher temperature difference between the heating medium and the foods causes faster heat penetration (Al-Baali and Farid, 2006). Hence, penetration of heat into the slowest heating zone (SHZ) (i.e., the location of the lowest temperature) in the food is controlled by thermal properties of the food itself (Al-Baali and Farid, 2006). The information provided by decimal reduction time (D_T) and F_o values are valuable, as they provide information on how fast a specific microorganism can be destroyed and also model the effect of temperature on the rate of destruction.

The effects of the independent variables on the F_0 of the sterilized soups are shown in (Tables 1) and (Figure 4). Table 1 shows sterilization temperature was the only factor (independent variable) that had significant ($p<0.05$) effect on the F_o values of the soups subjected to the thermo processing. As shown by the single factor plots in (Figure 4), soup and sterilization time had near plain horizontal line inclination ($\theta \approx 0°$) in the plot, suggesting a "no change" scenario in F_0 as the factor level changed, while F_0 showed clearly marked linear increase with increase in sterilization temperature. According to Kraber (2005) and Chakraborty *et al*. (2013), the inclination (steepness of the gradients) of the factor plot shows the magnitude of the effect of the factor, indicating that the

effect of the sterilization temperature was the most pronounced followed by the effect of sterilization time, while the soup type came least. From the coefficient estimates in Equation 4, it could be seen that, on the average, a unit change in the soup from egusi to ogbono caused an insignificant ($p>0.05$) reduction in the lethality property of the sterilization by 0.3697, while a unit change in the sterilization time increased the lethality by an equally insignificant ($p>0.05$) value of 1.86. Conversely, a unit increase in the level of sterilization temperature resulted in a significant ($p<0.05$) increase in the F_0 by 16.02. This therefore suggests why duration of sterilization (thermal processing time) is set following the chosen sterilization temperature and not the other way round. Hence, in setting thermal processing regimes and in computing lethality, the sterilization temperature usually appears to receive more consideration than time.

Heat treatment of the canned product is essential in relation to its safety and stability, and must be sufficient to ensure that no microbiological hazard arises during storage (Ostoja *et al*., 2002). Severe heat treatment during sterilization produces substantial changes in nutritional and sensory qualities of foods (Al-Baali and Farid, 2006). Beside the lethal effects on microbial populations, the possible destructive effects of thermal processes on enzymes, certain nutrients and other quality properties, including sensory and colour loss, have also been indicated by their F_0 and z values (Toledo, 2007). The z values of vitamins and colour loss are in the minimum two to three times higher than that required for microbial inactivation. This therefore suggests that from the 110 °C/90 minutes sterilization and above, where minimum botulinum cook could be attained, a possible severe degradation of these quality indices at rates double or more of the microbial inactivation could be expected. The temperatures reached during processing may affect radically the characteristics and composition of food by enhancing lipid oxidation and other processes (Secci and Parisi, 2016).

Conclusion

The study has shown that the indigenous soups, egusi and ogbono, canned in glass containers can have sufficient heat penetration for commercial sterility. However, to achieve this, the sterilization system would require a retorting temperature of 115 °C for a minimum of sixty minutes. The sterilization temperature is the most important variable to control, as the soup variety being canned and sterilized is immaterial. The F_0 of the soups can be used as a guide for selecting temperature-time combination for the sterilization of the studied indigenous soups in event of adoption of the technology at industrial level. The choice would depend on intended severity of sterilization. While increasing the degree and duration of sterilization would increase the safety level of the product, the use of 115 °C for duration not in excess of ninety minutes would achieve needed commercial sterility without fear of possible overcooking, which could be the case at higher temperatures.

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