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The Effect of Temperature on the Drying Characteristics of Salted Shark Fillets

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Abstract: *Shark fillets (10 x 5 x 1 cm) which were previously osmotically treated in saturated brine solution (4h, 30°C) were dried in a horizontal air flow cabinet oven at 40°C (40% rh), 50°C (25% rh) and 60°C (15% rh) as well as under tropical ambient conditions (30°C, 60% rh). The air speed in the oven was fixed at 1.5 m/s and under ambient laboratory conditions, less than 0.5 m/s. Sample weight, moisture content, salt content and water activity were measured as the slabs dried. Slab moisture content decreased logarithmically with drying time, and increasing the dry bulb temperature from 30°C to 50°C resulted in a greater decline in moisture content as well as lower equilibrium moisture values. Further increasing the temperature to 60°C resulted in an initial increase in drying rate, followed by a dramatic decline due to case hardening of the slabs. Drying of slabs at 30°C and 40°C occurred in both the constant rate and falling rate periods. Drying at 50°C and 60°C occurred in the falling rate period only. Rate constants for the first falling rate period (k_1) increased from 0.0389 to 0.2486 h^{-1} as drying temperature increased from 30-60°C. At the same temperatures, diffusion coefficients for the first falling rate period (D_1) calculated according to Fick's Law increased from 1.1 x 10^{-6} to 7.0 x 10^{-6} cm^2s^{-1}.*

Keywords: *Shark, Drying, Fick's Law, Drying Rate Constant, Diffusion Coefficient*

1. Introduction

Dried salted fish is a very popular food item for West Indians in the Caribbean, Europe and Canada, where the demand for dried salted fish is driven more for the flavour of the product than for preservation purposes. In an attempt to provide an acceptable alternative to the costly, imported salted codfish, as well as to create an alternative outlet for the less popular species, various species of fish have been used with variable success in the production of dried salted fish. In Trinidad and Tobago (T&T), shark is commonly used for this purpose.

Dehydration and salt (sodium chloride) absorption are the fundamental processes contributing to the stability of dried salted fish. Preservation of fish by salting and drying is achieved by lowering the water activity (a_w) of the fish flesh (Ismail and Wooton, 1992). However, drying techniques for salted fish are often haphazard and weather dependent as drying is most commonly carried out in direct sunlight. This contributes to the production of salted fish with inferior sensory characteristics and keeping quality.

Due to the problems associated with sun drying, consideration is being given to the use of solar dryers (Chavan et al., 2008; Reza et al., 2009; Kituu et al., 2010) and the mechanical dehydration of fish (Bellagha et al., 2002; Andres et al., 2005; Bellagha et al., 2006; Nooralabettu, 2008; Bras and Costa, 2010; Boeri et al., 2011). New studies on drying techniques include the use of a heat pump dehumidifier (HPD), low temperature and high velocity (LTHV) drying and microwave-assisted drying (Shi et al., 2008; Kilic, 2009; Mohd Rozainee and Ng, 2010).

The analysis of drying mechanisms and the mathematical modelling of food drying processes allow us to understand the process parameters that are under the control of the operator as well as to predict drying behaviour under given drying conditions. Diffusivity is used to indicate the flow of moisture out of material during drying and is considered to be a relevant transport property necessary for the correct modelling and understanding of the food drying process (Guine, 2006; Jain and Pathare, 2007). The study of the experimental kinetics and modelling of the mass transfer phenomenon in fish during air drying therefore continues to be of great interest (Park, 1998; Bellagha et al., 2002; Bellagha et al., 2006; Jain and Pathare, 2007; Chavan et al., 2008; Kituu et al., 2010; Boeri et al., 2011).

While there have been some early drying studies on local species of fish, these have not included any information on the diffusion phenomena. This work seeks to advance the knowledge of the drying behaviour of locally available fish and the objective of this study is to investigate and mathematically model the mass transfer changes during the air drying of salted shark fillets dried at different temperatures.

2. Materials and Methods

2.1 Raw material handling

Shark (*Carcharhinus leucas*, Muller and Henle) was obtained from a local supplier. Upon capture, fish were cleaned, gutted and skinned before being split in half and filleted. The fillets were transported to the Processing Laboratory, at The University of the West Indies, St. Augustine, in an iced box, where they were immediately cut into rectangular slabs (10cm x 5cm). These pieces were then carefully placed in reclosable plastic freezer bags and stored overnight in a freezer. The following day, fillets were thawed partially to allow for easy cutting, and accurately cut into 1cm thick slabs using a Hobart food slicer (Model 1612E).

2.2 Osmotic dehydration (Salting)

Saturated brine made from food-grade sodium chloride dissolved in distilled water was used as the osmotic medium. The brine solution and the samples were contained in temperature-controlled stainless-steel water baths with water circulators (BlueM Constant Temperature Waterbath, Model WB1110A, NC, USA). Ten litres of brine was used for each osmotic run and the ratio of fish slabs to brine was 1:20, high enough to avoid any significant concentration change in brine during salting ((Lazarides and Mavroudis, 1997; Andres et al., 2005; Mujaffar and Sankat, 2006). Brine concentration was checked periodically during each osmotic run and brine saturation was maintained by suspending a fine nylon mesh containing solid salt in the solution (Mujaffar and Sankat, 2006). The solution was constantly recirculated at an average flow rate of 200 ml/s to improve mass transfer.

Preliminary experiments revealed that salt gain and water removal both occurred at acceptable rates at ambient temperature (30°C) so this temperature was selected as a matter of convenience. Prior to osmotic dehydration, fillets were immersed for 5 min in a 5 % citric acid solution. This procedure is recommended as a bleaching step as well as to inhibit the conversion of urea to ammonia in shark. Fillets 1cm thick were salted for a period of 4 h. A 4 h immersion time was sufficient to allow adequate salt absorption (at least 12%) to stop normal bacterial spoilage (Mujaffar and Sankat, 2006). After brining, samples were quickly rinsed with water to remove any surface salt to reduce the appearance of white salt crystals on the surface during subsequent drying (Clucas and Sutcliffe, 1981).

2.3 Air (oven) drying

Drying of fillets was carried out in a constant temperature, horizontal air flow cabinet oven (BlueM Electric Company, Illinois, U.S.A.). The dryer speed in this oven was fixed at 1.5 m/s. Air velocity of the drying air was measured using a digital thermo-anemometer (EXTECH Instruments, U.S.A.) and relative humidity measured using a Humitemp monitor (Phys-Chem Scientific Corp, New York, U.S.A.). Fillets were spread in single layers on pre-weighed shallow wire mesh trays. Five samples were placed on each tray, and two trays were used for each drying run. At regular intervals, the samples were taken out, quickly weighed and returned to the dryer. Drying was continued for a maximum of 72 h, and after that samples were dried in an oven at 105°C for 24 h to determine dry mass. From this data, the average values of moisture content as a function of time were determined, and used to construct the drying curves (Sankat et al., 1996).

To evaluate the effect of air dry bulb temperature on the drying process, salted fillets (1cm thick) were dried at ambient laboratory temperature (30°C), and at three oven temperatures, 40°C, 50°C and 60°C. The relative humidity of the drying air under ambient conditions and at each of the three oven temperatures was 60, 40, 25 and 15 %, respectively. The air speed in the dryer was fixed at 1.5 m/s and under ambient conditions, less than 0.5 m/s.

2.4 Automated Erosion Wheel

Weight in grams (g) was measured using an Ohaus Electronic Balance (Ohaus Scale Europe Ltd., Cambridge, England). Moisture content, expressed both on a percentage fresh weight basis (g H_2O/100g FW) and on a dry weight basis (g H_2O/g DM), was determined by an oven-drying method (FAO 1981). Samples were dried for 24h at 105°C in a Gallenkamp Size One BS (Leicestershire, England). Salt (NaCl) content of the fillets was determined titrimetrically using silver nitrate solution (FAO, 1981) and expressed both as percentage salt on a fresh weight basis (g NaCl/100g fresh weight) as well as on a dry weight basis (g NaCl/g DM). Water activity (a_w) was measured using a water activity meter (Rotronic Hygroskop DT, Rotronic Instrument Corp., NY) and calculated as the equilibrium relative humidity divided by 100 (Gould and Gould 1988).

2.5 Automated Erosion Wheel

Data analysis consisted of simple regression analysis using Microsoft Excel 97 to examine the data for good fit. Further Regression Analysis and ANOVA were carried out using Genstat Statistical Software (Lawes Agricultural Trust, 1996).

3. Description of the Constructed Research Facility

3.1 Air drying curves

Air drying curves for salted shark fillets at 30°C to 60°C are shown in Figure 1. Moisture content was significantly affected by drying time, dry bulb temperature, and a time-temperature interaction ($p \leq 0.001$). The moisture content of untreated slabs prior to salting averaged 2.86 g H_2O/g DM (74.1% wet basis). After the osmotic treatment, this was reduced to 1.66 g H_2O/g DM (62.4% wet basis) and the salt content increased to 0.40 g NaCl/g DM (16% wet basis), respectively.

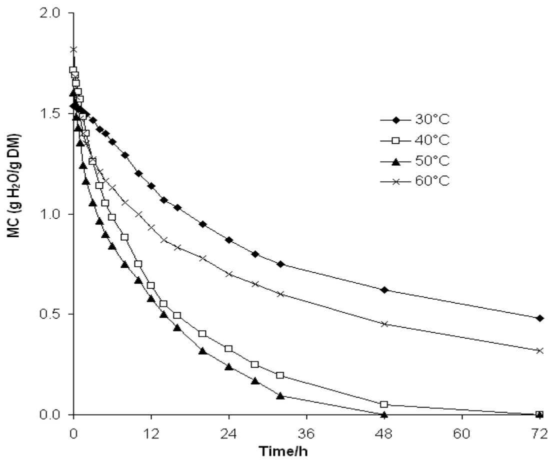

Figure 1. The effect of temperature on the moisture changes in salted shark slabs (10cm x 5 cm x 1cm) during air drying

Increasing the temperature from 30°C to 50°C resulted in a higher decline in moisture content with drying time, however further increasing the temperature to 60°C reversed this trend. Equilibrium moisture values are usually dependent upon the drying conditions, falling as the air dry bulb temperature is increased and the relative humidity decreased. This was found to be true in the present study as the temperature increased from 30°C to 50°C. Equilibrium moisture values were found to decrease dramatically from 0.48 g H_2O/g DM for salted slabs dried at 30°C to near zero for slabs dried at 40 and 50°C. Water activity values for salted slabs dried at 30, 40 and 50°C for 32h averaged 0.794, 0.772 and 0.436, respectively.

Salted slabs at 60°C appeared dried on the outside, but were still moist on the inside, a classic example of case hardening. Moisture and water activity values for salted slabs dried at 60°C for 32h averaged 0.30 g H_2O/g DM and 0.782, respectively. The dried crust on the slab surface was found to be 2-3 mm thick. Case hardening in fish is the progressive formation of a thick crust of salt and protein at the surface. Surface layers dry quickly, producing a hard layer which is impervious to the passage of water. This layer then prevents the migration of water and the centre of the fish can become spoiled although it looks well dried (Wheaton and Lawson, 1985).

Bellagha et al. (2006) attributed higher water content values in dry-salted whole sardines compared with brined sardines dried at 40°C to the formation of a crust which could be seen on the surface of the fish. Fernando et al. (2008) investigated case hardening in papaya and garlic slices and noted that drying of temperature sensitive food and agricultural products at high temperatures could lead to case hardening due to cell damage and gelatinization which restricts moisture movement and retards drying rates. A model for the constant temperature drying rate of case hardened slices of papaya and garlic was proposed.

Analysis of four commercially available brands of salted fish available locally revealed that the moisture content averaged between 0.83 to 0.89 g H_2O/g DM (44-47% wet basis) and water activity values 0.786 to 0.789. To achieve a final, safe moisture content of approximately 0.69g H_2O/g DM (or 40% wet basis), salted shark slabs can be dried for a minimum of 8h at 50°C, or a minimum of 10h at 40°C. It was noted that slabs dried at 50°C appeared to be drier and less pliable than those dried at 40°C to the same moisture content, and these characteristics were preferred.

3.2 Rate curves

Moisture content data was used to calculate the rate of change in moisture with drying time. This was done by calculating the difference in moisture content (g H_2O/g DM) between consecutive sampling times, and dividing this value by the time interval (h). The drying rate curves (rate versus drying time) for the air drying of salted shark slabs are shown in Figure 2.

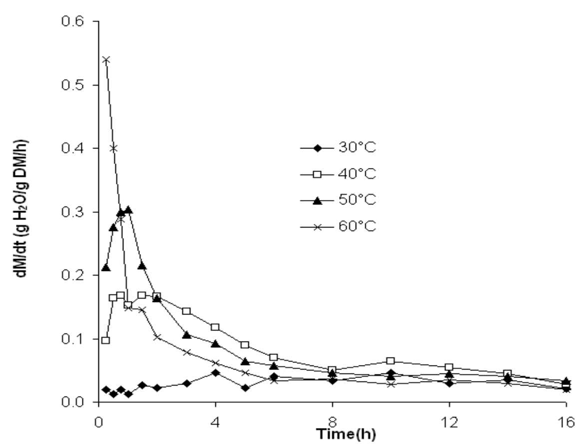

Figure 2. Plots of drying rate (dM/dt) versus time for salted shark slabs (10cm x 5 cm x 1cm) dried at different temperatures

The rate of change in moisture content was significantly affected by drying time and drying temperature ($p \leq 0.001$). Generally, with the exception of slabs dried at 30°C, drying rates were highest during the first 2h of drying, when the moisture content was greatest. After this time rates declined gradually. Drying rates for slabs held for 1.5h at 50°C were 50% higher than those dried at 40°C. After 5h, these rates declined to values 30% lower than the slabs at 40°C. Drying rates for slabs dried at 60°C were very high, averaging 0.54g H_2O/g DM/h. After the first hour of drying, drying rate declined drastically to values lower than those for other oven dried slabs.

The drying rate curves (rate versus average moisture content) for the air drying of salted shark slabs are shown in Figure 3. The rate of change in moisture content was significantly affected by moisture content and moisture content-temperature interaction ($p \leq 0.001$). Drying at 30 and 40°C occurred both in the constant rate and falling rate periods, while drying at 50 and 60°C occurred in the falling rate period only. For slabs at 30 and 40°C, there was a period of constant rate drying. Slabs dried at 30°C showed a constant rate of drying at 0.02-0.05 g H_2O/g DM/h. Slabs dried at 40°C showed a constant rate of drying for the first 2h, averaging 0.16 g H_2O/g DM/h.

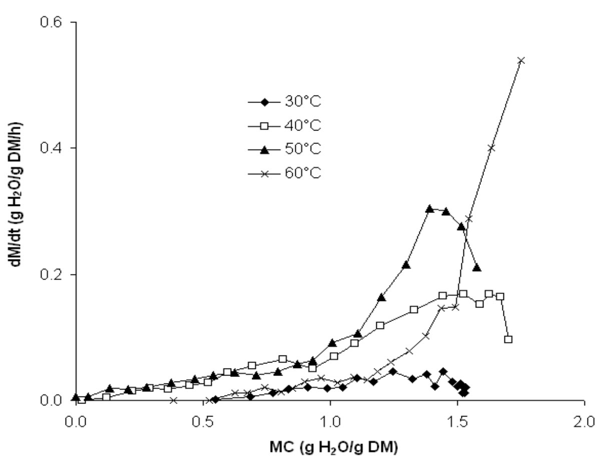

Figure 3. Plots of drying rate (dM/dt) versus average moisture content for salted shark slabs (10cm x 5 cm x 1cm) dried at different temperatures

After a certain critical moisture content (approximately 1.25 g H_2O/g DM for slabs at 30°C and 1.44 g H_2O/g DM for slabs at 40°C), the drying rate curves began to decline. For slabs at 50°C, there was an initial warm up period in which the rate of drying progressively increased, and then there was a rapid decline in rate, followed by a more gradual decline to equilibrium conditions in one or more linear portions. The drying rate of slabs at 60°C was initially high and declined drastically. This implies that internal water movement was controlling from the beginning of the drying process at these temperatures (Yusheng and Poulsen, 1988).

The constant rate drying under ambient conditions (30°C) and at 40°C was most likely due to the low drying rate potential of the air and the formation of a saturated layer of air above the sample surface (Chirife, 1983). Drying rate curves for whole brined sardines at 40°C (15% rh and 1.5 m/s) revealed that drying occurred in two falling rate periods and drying rates were shown to increase as the drying temperature increased from 35 to 50°C (Bellagha et al., 2002).

3.3 Drying constants

In food drying modeling, Fickian models are widely accepted and the determination of drying rate constants and moisture diffusivity often determined through analysis of the experimental drying data. It is generally accepted that moisture is removed from salted fish during the falling rate period during which the rate is governed by the transfer of water by diffusion (Ismail and Wooton, 1992; Park, 1998; Bellagha et al., 2002; Kituu et al., 2010; Boeri et al., 2011). The analytical solution of Fick's second law of diffusion for an infinite slab assuming uniform initial moisture distribution and negligible external resistances reduces to the straight-line equation for long drying times (Equation 1). The drying constant (k) is obtained from the slope of the plot of *ln* M_r versus *t*.

$$M_r = \frac{M - M_e}{M_o - M_e} = Ae^{-kt} \qquad \text{.....Eq.1}$$

where M_r = moisture ratio, M = moisture content (g H_2O/g DM), M_e = equilibrium moisture content (g H_2O/g DM), M_o = initial moisture content (g H_2O/g DM), k = rate constant or drying constant (h^{-1}), t = drying time (h).

To satisfy the condition that surface resistance to diffusion may be considered negligible, drying was carried out under forced convection. In many studies on the drying of fish, whole small fish are approximated as cylinders, and split fish are approximated as flat slabs (Bellagha et al., 2002; Chavan et al., 2008; Nooralbettu, 2008). However, arbitrary shapes as well as the presence of skin and fins can make model-based calculations difficult. The use of rectangular slabs as done in this study continues to be an important consideration in diffusion studies on fish and beef (Oladele and Odedeji; 2008; Kituu et al, 2010; Wafa et al., 2011).

When the experimental data for salted slabs were plotted as free moisture versus time based on Equation 1, Figure 4 was obtained. A straight line was obtained for salted slabs dried at 30°C, which means that the falling rate period following the constant rate period occurred in one phase. Moisture ratio plots for slabs dried at 40-60°C were broken into two linear portions, representing two falling rate periods. The first linear portion ended after 6h (0.98 g H_2O/g DM), 2h (1.16 g H_2O/g DM) and 1.5h (1.40 g H_2O/g DM) for slabs dried at 40, 50 and 60°C, respectively.

Uddin et al. (1998) reported that for the drying of unsalted *Alute mate* fish samples (4cm x 4cm x 1.2cm) at 40 to 70°C, no constant rate period was observed, and a second falling rate period was present in all cases. The second falling rate period started at a moisture content of approximately 2.1 g H_2O/g DM.

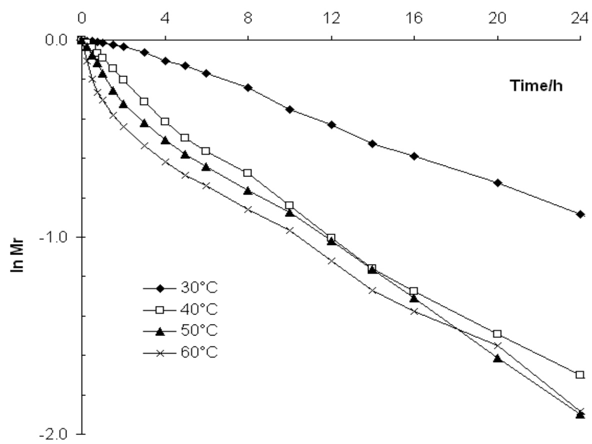

Figure 4. Plots of free moisture (moisture ratio) for salted shark slabs (10cm x 5 cm x 1cm) dried at different temperatures

Bellagha et al. (2002) noted that several studies show two falling rate periods when drying salted fish, but for most industrial processes, drying is stopped before the beginning of the second falling rate period. The transition from the first to the second falling rate phase in cod muscle during air drying corresponds with the uncovering of the monomolecular inner layer of water molecules after the multiple-molecule outer layer is removed (Peters, 1971; Bellagha et al., 2002).

Linear regression was employed to obtain values of rate constants for different dry bulb temperatures. The data for slabs dried at 30°C was successfully fitted with one straight line characterised by a drying constant, k_1. The data for salted slabs dried at 40-60°C was fitted with two straight lines each characterised by a drying constant, k_1 and k_2. Drying constants are given in Table 1. The k-values for the first phase were always higher than those for the second.

Increasing the drying temperature from 30°C to 60°C significantly enhanced the drying rate and the k_1-values increased from 0.0389 to 0.2486 h^{-1}. Values of rate constants were 1.7 times higher at 50°C than at 40°C.

Table 1. The effect of drying temperature on the drying rate constants (k) of salted shark fillets (10 x 5 x 1cm)

Temperature (°C)	rh (%)	Drying Rate Constants (h^{-1})			
		k_1	r^2	k_2	r^2
30	60	0.0389	0.9982	-	-
40	30	0.0993	0.9961	0.0645	0.9906
50	25	0.1660	0.9972	0.0715	0.9979
60	10	0.2486	0.9438	0.0637	0.9970

However, for slabs dried at 60°C the first falling rate period was very short (1.5h), and the k_2-values were considerably lower than the k_1-values, a reflection of the drastic decrease in drying rate due to case hardening during the initial stages of drying. An increase in rate constant with increasing drying air temperature has been shown for the drying of many biological materials (Chiang and Petersen, 1985; Yusheng and Poulsen, 1988; Sankat et al., 1996; Krokida et al., 2004). Sankat and Mujaffar (2004) found the k_1-values for salted shark slabs dried in open sun, direct and indirect solar cabinet dryers to be 0.1381, 0.1264 and 0.0838 h^{-1}, respectively. Rate constant data reported for salted fish is found to be lower than that reported for unsalted fish. This is in agreement with the generally accepted fact that the higher the salt concentration, the more slowly the fish dries. Jain and Pathare (2007) reported an average rate constant of 0.2063 h^{-1} for unsalted sun dried carp (32.5 - 42.5°C, 15 - 32% rh).

3.4 Diffusion Coefficients

The drying rate constants for unsalted and salted slabs were used to calculate diffusion coefficients using the following equation according to Equation 2:

$$k = \pi^2 D / 4L^2 \qquad \text{.....Eq.2}$$

where L is one half the thickness of the slab.

Diffusion coefficients for the first falling rate period (D_1) for slabs increased with increasing drying temperature. In the present study, D_1-values for salted shark slabs averaged 1.09, 2.81, 4.67, and 7.00 x 10^{-6} cm^2s^{-1} for slabs dried at 30, 40, 50 and 60°C, respectively.

Diffusion coefficient values of 2.6 x 10^{-6} and 3.3 x 10^{-6} cm^2s^{-1} have been reported in the literature for salted swordfish slices (0.6cm thick, with skin) dried at 40°C and 55°C, respectively (Chirife, 1983). Boeri et al. (2011) found the D_1-values for salted whole codfish dried at 20°C under different conditions of air velocity and relative humidity to range from 2.71 to 4.39 x 10^{-6} cm^2s^{-1}. Boeri et al. (2011) also quoted the results of previous work with diffusion coefficients ranging from 1.25 to 3.27 x 10^{-6} cm^2s^{-1} for salted fish fillets.

The effect of temperature on the diffusion coefficient is to increase the rate of diffusion as temperature increases (Ismail and Wooton, 1992). Park (1998) found that increasing the drying temperature from 20 to 40°C resulted in an increase in D_1-values for salted shark pieces from 1.50 to 2.85 x 10^{-6} cm^2s^{-1}. Values of effective diffusivity for the drying process taking into account shrinkage of fish muscle were approximately 40% lower.

For salted slabs dried at 40, 50, and 60°C, D_2-values were lower than the D_1-values, and averaged 1.81, 2.01, and 1.80 x 10^{-6} cm^2s^{-1}, respectively. D_2-values reported in the literature for fish dried at 30°C range from 0.35 to 0.81 x 10^{-7} cm^2s^{-1}, respectively (Jason, 1958). According to Jason (1980), the rate of diffusion is always lower for the second falling rate period because the activation energy at this stage is higher.

3.5 Arrhenius Plots

The temperature dependence of the D-values for unsalted and salted slabs and the activation energies were estimated from a plot of ln D versus 1/T (see Figure 5) using an Arrhenius type equation:

$$\ln D = -\frac{E_a}{RT} \quad \text{.....}\mathit{Eq.3}$$

where E_a = activation energy ($Jmol^{-1}$), R = gas constant (8.314 $JK^{-1}mol^{-1}$), T= process temperature (K).

The activation energy for the first falling rate period for salted fish slabs dried at 30 to 60°C was calculated to be 51.1 KJ/mol (r^2 = 0.9729). Park (1998) reported an activation energy of 21.9 KJ/mol for salted shark pieces dried at temperatures ranging from 20 to 40°C.

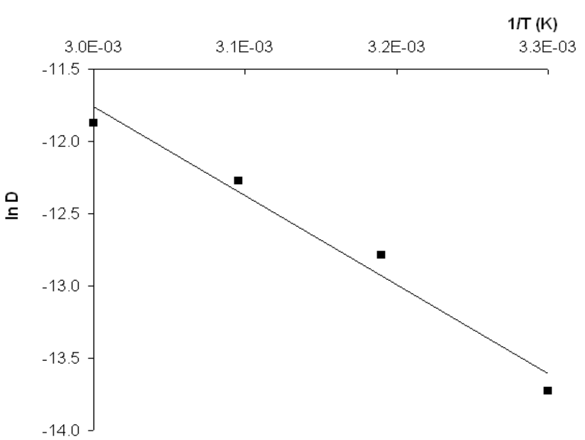

Figure 5. Relationship between moisture diffusion coefficient (D) and drying temperature for salted shark slabs (10cm x 5 cm x 1cm)

4. Conclusions

Salted shark fillets dried at 30-50°C show the typical decline in moisture content with drying time, with the greatest decline occurring during the initial stages of drying. Further increasing the temperature to 60°C results in case hardening of the slabs. Drying at ambient temperature (30°C) and at 40°C occurs in both the constant rate and falling rate periods. Drying at 50°C and 60°C occur in the falling rate period only. The drying data can be modelled using the analytical solution for Fick's Law for an infinite slab assuming uniform initial moisture distribution and negligible external resistances. The falling rate period for salted fish fillets dried at 40-60°C occur in two phases, each characterised by a drying rate constant (k_1 and k_2). Drying rate constants for the first phase (k_1) are higher than those for the second phase (k_1). Both the drying rate constants and diffusion coefficients in the first falling rate period increase with increasing temperature.

A final, safe moisture content of approximately 0.69g H_2O/g DM (or 40% wet basis) can be achieved in salted shark slabs (10 x 5 x 1cm) which have been osmotically treated in saturated brine for 4h at room temperature, and then oven dried under forced convection for 10h at 40°C or 8h at 50°C. The higher k_1 and D_1-values in slabs dried at 50°C and the favoured appearance and texture of the slabs make drying at 50°C the preferred drying temperature for commercial salted fish production.

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