

# **PREDICTION OF WATER SORPTION PROPERTIES OF EXTRUDED ACHA/SOYBEAN BLENDS USING RESPONSE SURFACE ANALYSIS**

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## **ABSTRACT**

Water sorption isotherms of raw and extrudate samples of acha/soybean were constructed at ambient temperature ( $27\pm 2^\circ\text{C}$ ) with saturated salt solutions of known water activities. Equilibrium moisture content (EMC) was evaluated and monolayer values were determined following a four variable response surface analysis using central composite rotatable design that was nearly orthogonal. The results were fitted using multiple regression analysis. Model equations were developed from polynomials with significant model ( $P\leq 0.05$ ) differences. The EMC and monolayer values of extrudates were predicted using the developed models. The effects of extrusion variables (feed composition, feed moisture content screw speed and barrel temperature) on extrudate water sorption properties were also evaluated. Results showed that extrudates exhibited normal sigmoid shaped moisture sorption isotherms. At lower (0.35-0.40 g  $\text{H}_2\text{O}/100\text{g}$ ) water activities, the quadratic effect of feed moisture content (FMC) had the most significant ( $P\leq 0.05$ ) effect while the linear and cross product effects were not significant ( $P\geq 0.05$ ). At intermediate (0.6 g  $\text{H}_2\text{O}/100\text{g}$ ) water activity all process variables with the exception of feed composition (FC) exhibited significant ( $P\leq 0.05$ ) linear and cross product effects. At higher (0.75-0.90 g  $\text{H}_2\text{O}/100\text{g}$ ) water activities, the process variables exerted significantly, linear, cross product and quadratic effects, with the exception of feed composition. The monolayer values varied between 0.6-6.0 (g  $\text{H}_2\text{O}/100\text{g}$ ) but showed no significant ( $P\geq 0.05$ ) model difference. Predicted equilibrium moisture of extrudates was close approximation of experimental values. The feed moisture content and screw speed exerted a significant ( $P\leq 0.05$ ) influence on extrudate equilibrium moisture content during the experimental period. Monolayer values showed that extrudates would remain chemically and microbially stable at ambient temperatures where longer storage period is intended.

**KEYWORDS:** Equilibrium moisture, Response surface, Blends, Variables and Extrudates.

## **INTRODUCTION**

Acha (*digitaria exilis skippis staph*) also known as fonio or hungry rice is a cereal of unique characteristics (Kwon-dung and Misari, 2000). According to Jean-Francois (2004) acha is the world's best tasting cereal having greater methionine and cysteine content in their amino acid profile above the recommended values. It is also high in digestible energy but low in oils and minerals and is used in dietary application for diabetic patients (Victor and James, 1991). Soybean (*Glycine max L Merrill*) on the other hand is a versatile pulse, which constitutes the staple food in many parts of the globe. With high protein value and high polyunsaturated fat content with the absence of cholesterol and lactose, soybean is an excellent source of essential amino acid vital for body growth maintenance and reproduction (Iwe, 2003). Blending acha and soybean therefore would produce products with balanced nutrients. For retention of vital nutrients and development of variety of products a flash processing system like extrusion cooking offers the best option.

Extrusion cooking is a continuous, high capacity, sanitary, space and energy efficient and cost effective technology (Harper, 1981). It is highly versatile and adaptable. Its versatility is manifested in its wide spread application as a transporting, guiding, hydrating, shearing and forming instrument which is also used in destroying micro organisms, denaturing hydrolytic enzymes and inactivating antinutritional factors in food stuffs (ITF, 1989). According to FAO, (1985) and Iwe (2001) cereals including wheat, maize, sorghum and millet had been blended and extrusion cooked with soybean to produce complementary foods.

Sunday (2002) reported that knowledge of the water sorption characteristics of a food product was necessary in prediction of shelf life and determination of critical moisture and water activity for acceptability of products that deteriorate mainly by moisture gain such as cookies and snacks. Water content and its distribution govern the shelf life of baked products that are

influenced by incidence of microbial damage (Czuchajowska, 1989). However Sunday (2002) had shown that it is water activity (aw) rather than water content that determines the microbial spoilage of foods. There are presently no reports on the extrusion cooking of neither acha nor its blending with soybeans. The major focus of this present work was to develop predictive models that relate the equilibrium moisture content of extruded acha/soybean extrudates to the extrusion process variables. It was also to provide information on the influence of these variables on the sorption isotherms of acha/soybean extrudates.

## **MATERIALS AND METHODS**

**FEED PREPARATION:** The combination of independent variables is shown in Table 1. Acha and soybean flour were prepared for extrusion as shown in figure 1. Soybean flour was added to acha flour at 0, 12.5, 25, 37.5 and 50% levels (Table 1). Feed moisture content was adjusted at five levels of 15, 20, 25, 30 and 35% according to Wilmot, (1998). The transformed matrix showing the extrusion outlay and extruder condition is shown in Table 2.

**EXTRUSION:** Extrusion was carried out using a Branbender Laboratory single screw extruder (DUISBURG DCE - 330 Model). It was powered by a decoder drive (Type 832, 500) and driven by a 5.94kw motor. The grooved band had a length / diameter ratio of 20:1. The extruder had variable screws and heaters with a fixed die diameter of 2 mm and length of 40 mm. A feed hopper mounted vertically above the end of the extruder and equipped with a screw that rotated at a constant speed of 80 rpm on a vertical axis takes feed into the extruder. The screw speed was adjusted from 90, 120, 150, and 180 to 210 rpm while the die temperature was adjusted from 100, 125, and 150, 175 to 200°C as shown in Table 2. The wet flour was allowed to equilibrate for 2-3 hrs before extrusion. The extruder runs were stabilized using acha flour. Extrusion of the blends was then carried out as shown in the transformed

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matrix (Table 2).

**EXPERIMENTAL DESIGN:** The experimental design was a response surface analysis using a central composite rotatable design (CCRD). This design has a factorial, an axial and centre axis (points). Four independent variables including feed composition (FC), feed moisture content (FMC), screw speed (SS) and barrel temperature (BT) were tested at 5 levels coded (-2 to +2) as shown in Table 1 (Meyers, 1976 and Iwe, 2003). This experimental design required 36 extrusion runs of which 16 were performed at the factorial points, 8 at the axial point and twelve at the centre points (table 2).

Water sorption isotherms of extrudate samples were determined as described by Mohammed *et al.*, (1986). Saturated salt solutions for the required humidity's were prepared by saturating the salts in 20ml-distilled water at ambient (27±4 °C). The salts used were calcium chloride (CaCl<sub>2</sub>), Potassium carbonate (K<sub>2</sub>CO<sub>3</sub>), Sodium nitrate (NaNO<sub>3</sub>), Sodium chloride (NaCl) and Barium chloride (BaCl<sub>2</sub>). These were used to prepare water activities of 0.35, 0.40, 0.64, 0.75, and 0.90 g H<sub>2</sub>O/100g respectively.

Extrudate samples (1g) were placed in a desiccator containing the appropriate solution at ambient temperature (27±4 °C). The samples were weighed daily using a digital balance. Equilibrium condition was considered when two consecutive weightings gave a constant value. The moisture content of the equilibrated samples was determined according to AOAC

(1984).

The sorption isotherm was obtained by plotting the moisture content of the extrudates against water activity. The monolayer moisture values of the extrudates were calculated deploying the method of Labuza (1968).

**STATISTICAL ANALYSIS**

All results were subjected to standard statistical analysis. The generalized regression model fitted was  $Y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_4x_4 + b_{11}x_1^2 + b_{22}x_2^2 + b_{33}x_3^2 + b_{44}x_4^2 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{14}x_1x_4 + b_{23}x_2x_3 + b_{24}x_2x_4 + b_{34}x_3x_4 + \epsilon$  where Y= objective response X<sub>1</sub> = feed composition X<sub>2</sub> = feed mixture content X<sub>3</sub> = extruder screw speed and X<sub>4</sub>= extruder barrel temperature and ε= random error in which the linear, quadratic and interaction effects were involved using a step wise multiple regression analysis according to Howard, G. S (1983) and Mullen and Ennis (1979). The resulting models were tested for significance using analysis of variance (ANOVA) and coefficient of determination (R<sup>2</sup>). Significant terms were accepted at P≤ 0.05 (Jin *et al.*, 1994, Howard, 1983). The R<sup>2</sup> of 0.6 was accepted for predictive purposes. The terms that were not significant were deleted from the model equations. For each significant model equation, response surfaces in three dimensional plots were generated on a computer programme (Statistica) by holding the two variables with the least and second least effects on the response constant (centre points) and changing the other variable.

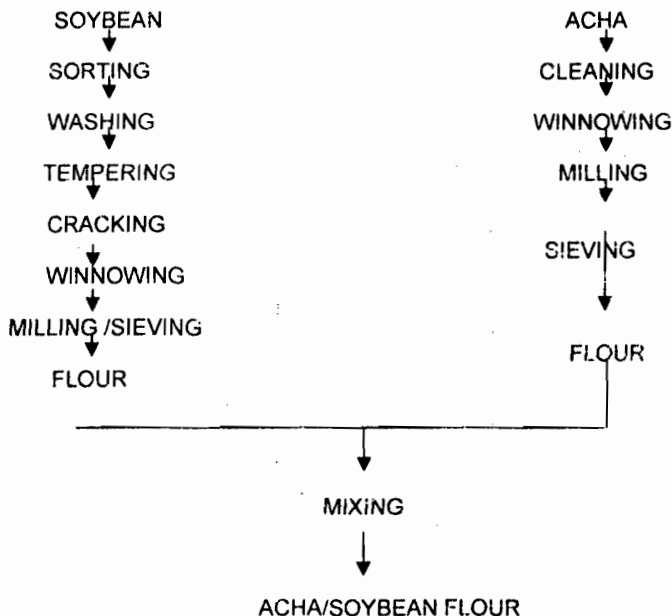


Fig 1: Preparation of Acha, Soybean Flours/Blends

**TABLE 1: Experimental Layout**

| Independent Variables   | Levels Of Combinations |      |     |      |     |
|-------------------------|------------------------|------|-----|------|-----|
|                         | -2                     | -1   | 0   | 1    | 2   |
| Feed Composition (%)    | 0                      | 12.5 | 25  | 37.5 | 50  |
| Feed Moisture (%)       | 15                     | 20   | 25  | 30   | 35  |
| Screw Speed (RPM)       | 90                     | 120  | 150 | 180  | 210 |
| Barrel Temperature (°C) | 100                    | 125  | 150 | 175  | 200 |

-2, -1, 0, 1, and 2= Coded levels of combination

**Results and Discussion:** The EMC of the raw samples ranged from 8.33 –9.52 g H<sub>2</sub>O/100g for acha flour and soybean flour and mixtures of acha and soybean flour respectively at 0.35 aw. It ranged from 8.48-13.64 g H<sub>2</sub>O/100g at 0.40 aw and 9.09-15.00 at 0.64 g H<sub>2</sub>O/100g aw. At 0.75, aw the range was from 11.35-15.00 g H<sub>2</sub>O/100g. The EMC at 0.90 aw was discarded because of molding. The results showed that blending resulted in increased EMC at all water activities tested. This was expected because increase in proximate composition is known to result to corresponding increases in water absorption capacity and solubility index due to changes in the protein value of the samples. Philip *et al* (1988) reported that water absorption of cowpea meals was related more to protein and starch composition than to particle size distribution.

Extrusion cooking reduced the EMC content of the extrudates and this was significantly ( $P \leq 0.05$ ) lower than the raw blend. Effects of the extrusion-processing variables on the sorption isotherms of extrudates are presented in tables 3-8. The estimated regression coefficients and anova analysis for EMC at 0.35aw is shown in Table 3. The results showed that at this aw, the linear and cross product effects of the independent variables did not exert significant ( $P > 0.05$ ) effect. The quadratic effect of FMC was however significant ( $P \leq 0.05$ ). Analysis of variance showed a model significance ( $P \leq 0.05$ ) indicating that the model fitted the linear regression. Removing the insignificant terms the resulting model equation became

$EMC(0.35) = 3.12 - 3.37FMC^2 - 1.73FC \cdot FMC - 1$   
 The response surface plot of  $FMC \cdot FC$  and EMC is shown in (Fig 2). The result showed that increasing FMC and FC had a quadratic effect on extrudates EMC at 0.35aw. The implication of this trend was that extruding at low feed moisture (15-20%) content resulted to highly expanded extrudates having many active sorptive sites for water binding. Conversely at high feed moisture content (30-35%) and increased substitution of soybean in the feedstock extrudates with reduced water sorption capacity were produced.

Table 2: Matrix Transformation of the Experimental Design Runs and Extrusion Conditions

|    | Feed Composition (g soybean) | Feed Moisture Content (%) | Screw Speed (rpm) | Barrel Temperature (°C) |     |          |
|----|------------------------------|---------------------------|-------------------|-------------------------|-----|----------|
|    |                              |                           |                   | 1                       | 2   | Die Temp |
| 1  | 125                          | 20                        | 120               | 125                     | 125 | 125      |
| 2  | 125                          | 20                        | 120               | 125                     | 125 | 175      |
| 3  | 125                          | 20                        | 180               | 125                     | 125 | 125      |
| 4  | 125                          | 20                        | 180               | 125                     | 125 | 175      |
| 5  | 125                          | 30                        | 120               | 125                     | 125 | 125      |
| 6  | 125                          | 30                        | 120               | 125                     | 125 | 175      |
| 7  | 125                          | 30                        | 180               | 125                     | 125 | 125      |
| 8  | 125                          | 30                        | 180               | 125                     | 125 | 175      |
| 9  | 375                          | 20                        | 120               | 125                     | 125 | 125      |
| 10 | 375                          | 20                        | 120               | 125                     | 125 | 175      |
| 11 | 375                          | 20                        | 180               | 125                     | 125 | 125      |
| 12 | 375                          | 20                        | 180               | 125                     | 125 | 175      |
| 13 | 375                          | 30                        | 120               | 125                     | 125 | 125      |
| 14 | 375                          | 30                        | 120               | 125                     | 125 | 175      |
| 15 | 375                          | 30                        | 180               | 125                     | 125 | 125      |
| 16 | 375                          | 30                        | 180               | 125                     | 125 | 175      |
| 17 | 500                          | 25                        | 150               | 125                     | 125 | 150      |
| 18 | 000                          | 25                        | 150               | 125                     | 125 | 150      |
| 19 | 250                          | 15                        | 150               | 125                     | 125 | 150      |
| 20 | 250                          | 35                        | 150               | 125                     | 125 | 150      |
| 21 | 250                          | 25                        | 90                | 125                     | 125 | 150      |
| 22 | 250                          | 25                        | 210               | 125                     | 125 | 150      |
| 23 | 250                          | 25                        | 150               | 125                     | 125 | 100      |
| 24 | 250                          | 25                        | 150               | 125                     | 125 | 200      |
| 25 | 250                          | 25                        | 150               | 125                     | 125 | 150      |
| 26 | 250                          | 25                        | 150               | 125                     | 125 | 150      |
| 27 | 250                          | 25                        | 150               | 125                     | 125 | 150      |
| 28 | 250                          | 25                        | 150               | 125                     | 125 | 150      |
| 29 | 250                          | 25                        | 150               | 125                     | 125 | 150      |
| 30 | 250                          | 25                        | 150               | 125                     | 125 | 150      |
| 31 | 250                          | 25                        | 150               | 125                     | 125 | 150      |
| 32 | 250                          | 25                        | 150               | 125                     | 125 | 150      |
| 33 | 250                          | 25                        | 150               | 125                     | 125 | 150      |
| 34 | 250                          | 25                        | 150               | 125                     | 125 | 150      |
| 35 | 250                          | 25                        | 150               | 125                     | 125 | 150      |
| 36 | 250                          | 25                        | 150               | 125                     | 125 | 150      |

Decreasing the FMC up to 20 % (fig 2) significantly ( $P \leq 0.05$ ) reduced extrudate EMC. Decreasing the level of soybean flour from 37.5% to 12.5% significantly ( $P \leq 0.05$ ) increased EMC and agree with the observation that at low and intermediate water activities, water is mainly held by physical absorption on polymeric compounds. In this study decrease in feed substitution led to significant increase porosity which resulted to reduced adsorption capacity. The results (Appendix 1) showed that minimal EMC was 6.00 (g H<sub>2</sub>O/100g) and this was obtained at 25% FC, 35% FMC 150 rpm 150°C TP. The

minimal predicted EMC was however 5.59 (g H<sub>2</sub>O/100g) at the same extrusion conditions. The maximal EMC was 10.54 (g H<sub>2</sub>O/100g) and was obtained at 25% FC, 25% FMC, 150 rpm and 150°C BT. The maximal predicted EMC was 10.60 (g H<sub>2</sub>O/100g) and was obtained at 12.5%FC, 30%FMC 180rpm and 175°C BT. A similar trend was observed for the extrudate EMC at 0.40 aw (Table 4). The results showed that only the quadratic effect of FMC significantly ( $p < 0.05$ ) affected the EMC at 0.40 aw.

TABLE 3: Estimated Regression Coefficients and ANOVA for EMC at 0.35aw

| Regression On Constants | Coefficients | Standard Error | P-Values | R <sup>2</sup> |
|-------------------------|--------------|----------------|----------|----------------|
|                         | 3.12         | 1221.40        |          |                |
| FMC                     | 2.44         | 1.66           | 0.68     | .62            |
| SS                      | 1.28         | 0.27           | 0.82     |                |
| BT                      | 0.15         | 0.28           | 0.98     |                |
| FC*FC                   | 1.12         | 3.15           | 0.13     |                |
| FMC*FMC                 | -3.37        | 0.01           | 0.02     |                |
| SS*SS                   | -2.02        | 2.00           | 0.12     |                |
| BT*BT                   | -0.58        | 2.88           | 0.70     |                |
| FC*FMC                  | -1.73        | 1.54           | 0.08     |                |
| FC*SS                   | 0.36         | 2.57           | 0.70     |                |
| FC*BT                   | 0.11         | 2.56           | 0.90     |                |
| FMC*SS                  | -0.66        | 0.01           | 0.93     |                |
| FMC*BT                  | 0.62         | 0.01           | 0.93     |                |
| SS*BT                   | -0.88        | 0.01           | 0.90     |                |
| FMC*SS*BT               | 2.98         | 6.64           | 0.73     |                |
| FC*FMC*SS*BT            | -0.04        | 6.80           | 0.97     |                |
| ANOVA                   | DF           | SS             | MS       |                |
| REGRESSION              | 15           | 30.50647       | 2.033761 |                |
| RESIDUAL                | 20           | 18.52499       | 0.92625  |                |
| F 2.19570               | SIGN. F 0.05 |                |          |                |

DF=Degree of freedom FC= Feed composition FMC= Feed moisture content  
 SS= Sums of Square S\*S= Screw speed  
 MS=mean square BT= Barrel temperature

There was a significant ( $P \leq 0.05$ ) model difference showing that the model fitted the linear regression. With acceptable coefficient of determination, ( $R^2=0.64$ ) the model was accepted for predictive purpose. Removing the insignificant terms in the regression, the resulting model equation became

$EMC (0.40 aw) = -16.40 - 2.94 FMC^2 + 8.88FMC$  -----Eq2.  
 The response surface plot of FMC\* SS and EMC is shown in Fig 3. The result showed that decreasing the FMC led to decreases in extrudate EMC as was noted at 0.35aw.

TABLE 4: Estimated Regression Coefficients and ANOVA for EMC at 0.40aw

| Regression On Constants | Coefficients | Standard Error | P-Values | R <sup>2</sup> |
|-------------------------|--------------|----------------|----------|----------------|
|                         | -16.40       |                |          |                |
| FMC                     | 8.88         | 1.36           | 0.13     | .64            |
| SS                      | 4.84         | 0.22           | 0.39     |                |
| BT                      | 2.38         | 0.23           | 0.63     |                |
| FC*FC                   | 0.59         | 2.57           | 0.41     |                |
| FMC*FMC                 | -2.94        | 0.04           | 0.03     |                |
| SS*SS                   | -0.11        | 1.64           | 0.93     |                |
| BT*BT                   | 1.07         | 2.36           | 0.48     |                |
| FC*FMC                  | -1.08        | 1.25           | 0.24     |                |
| FC*SS                   | -0.23        | 2.01           | 0.80     |                |
| FC*BT                   | 0.55         | 2.01           | 0.53     |                |
| FMC*SS                  | -9.62        | 0.01           | 0.29     |                |
| FMC*BT                  | -8.67        | 0.01           | 0.23     |                |
| SS*BT                   | -6.27        | 0.00           | 0.35     |                |
| FMC*SS*BT               | 11.85        | 5.43           | 0.16     |                |
| FC*FMC*SS*BT            | -0.03        | 5.55           | 0.97     |                |
| ANOVA                   | DF           | SS             | MS       |                |
| REGRESSION              | 15           | 22.44977       | 1.49665  |                |
| RESIDUAL                | 20           | 12.36260       | 0.61813  |                |
| F 2.42126               | SIGN. F 0.03 |                |          |                |

DF=Degree of freedom FC= Feed composition FMC=Feed moisture content  
 SS= Sums of Square S\*S= Screw speed  
 MS=mean square BT= Barrel temperature

EMC  
(0.35)

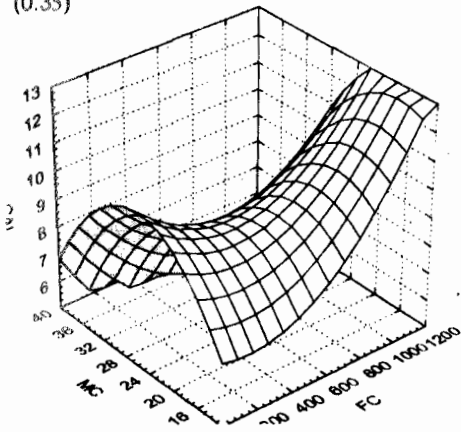


Fig 2: Response plot of EMC at 0.35 with FMC & FC

EMC  
(0.40)

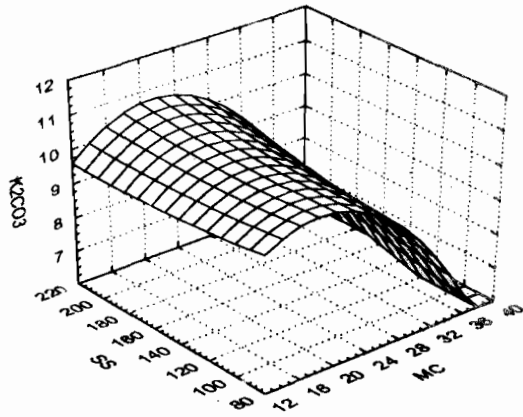


Fig 3: Response plot of EMC at 0.40 with FMC & SS

EMC  
(0.64)

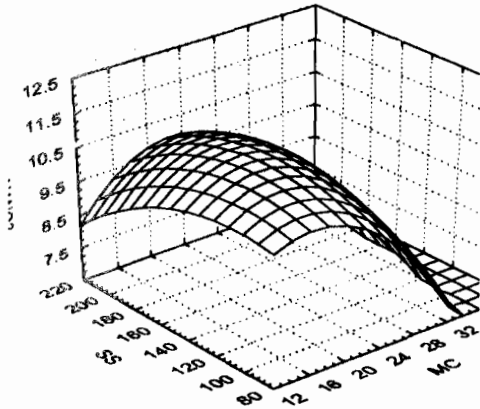


Fig 4: Response plot of EMC at 0.64 with FMC & SS

EMC  
(0.64)

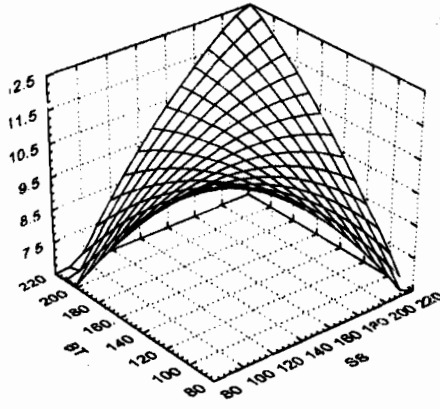


Fig 5: Response plot of EMC at 0.64 with SS and BT

EMC  
(0.64)

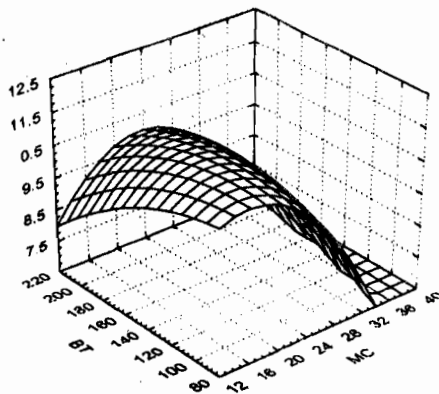


Fig 6: Response plot of EMC at 0.64 with FMC & BT

The result of the EMC at 0.64 aw is shown in Table 5. All the processing variables with the exception of FC exerted significant ( $P < 0.01$ ) linear effects on extrudate equilibrium moisture content. The cross product effects of FMC\*SS, FMC\*BT and SS\*BT were also significant ( $P \leq 0.01$ ). Analysis of variance showed a high significant ( $P \leq 0.0001$ ) model difference with a high coefficient of determination ( $R = 0.84$ )

indicating that the model adequately fitted the experimental data. The resulting polynomial after removing the non significant terms became  

$$\text{EMC at 64\% humidity} = -57.80 + 21.10 \text{ FMCSS} + 3.12 \text{ FMC}^2 + 13.54 \text{ FMC} - 16.68 \text{ FMCSS} - 15.65 \text{ FMC} \text{ TP} - 12.80 \text{ SS} \text{ TP} + 10.80 \text{ SS} + 8.62 \text{ TP} - 1.55 \text{ SS}^2$$
 Eq 3

EMC  
(0.75)

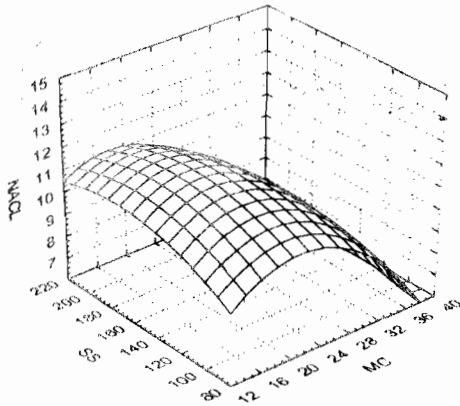


Fig 7: Response surface plot of EMC at 0.75 with SS & FMC

EMC  
(0.75)

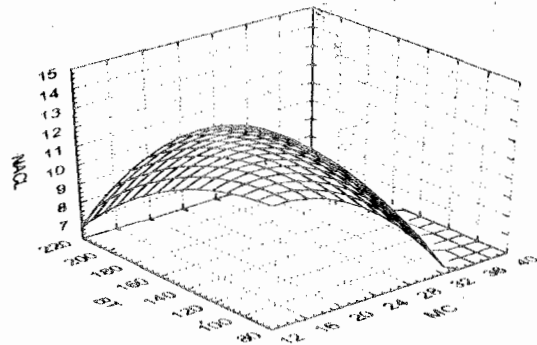


Fig 8: Response plot of EMC at 0.75 with FMC & BT

EMC  
(0.90)

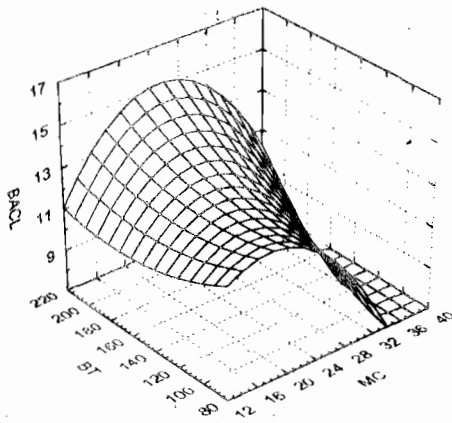


Fig 9: Response plot of EMC at 0.90 with FMC & BT

EMC  
(0.90)

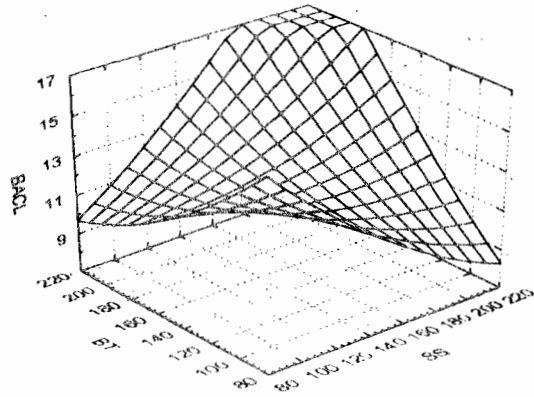


Fig 10: Response plot of EMC at 0.90 with BT & SS

The response surface relating EMC to FMC and SS is shown in Fig. 4. The surface showed that increasing the FMC and SS beyond 25% and 175 rpm led to decreased EMC. This decrease was observed from 20 to 32% FMC and 180 rpm. This phenomenon could be explained from the fact that increased moisture content favoured greater starch gelatinization and hence greater starch damage. Higher SS favoured greater shearing and higher dissipation of heat thus increased BT leading to greater gelatinization and greater starch damage. However, Czuchajowska *et al* (1989) reported that moisture and water activity generally were low in baked bread crust in which the starch was damaged. Emmanuel (2004) observed similarly that increased starch damage

resulted in lowered solubility of maize meal due to immobilization of starch within the granule during cooking. The results obtained in this work showed that greater starch damage at higher FMC and SS led to decreased EMC values. A similar trend was observed for the plot of EMC, FMC and BT (Fig 5)

The plot of EMC, extruder BT and SS (Fig 6) showed that increasing the SS beyond 120 rpm and BT 175°C led to decreased EMC. The results showed that EMC as low as 7gH<sub>2</sub>O was obtained at 180rpm Screw speed and 175°C. Elevated temperatures and high SS encourage greater starch damage resulting in lowered adsorptive capacity. increased

TABLE 5: Estimated Regression Coefficients and ANOVA for EMC at 0.64aw

| Regression On | Coefficients | Standard Error | P-VALUES | R <sup>2</sup> |
|---------------|--------------|----------------|----------|----------------|
| Constants     | -57.80       |                |          |                |
| FMC           | 13.54        | 0.96           | 0.00     | .84            |
| SS            | 10.80        | 0.16           | 0.01     |                |
| BT            | 8.62         | 0.17           | 0.01     |                |
| FC*FC         | 0.11         | 1.82           | 0.82     |                |
| FMC*FMC       | -3.12        | 0.00           | 0.00     |                |
| SS*SS         | -1.55        | 1.15           | 0.07     |                |

|              |          |          |         |
|--------------|----------|----------|---------|
| BT*BT        | -1.01    | 1.66     | 0.31    |
| FC*FMC       | 40       | 8.85     | 0.51    |
| FC*SS        | 1.29     | 1.48     | 0.63    |
| FC*BT        | -0.06    | 1.48     | 0.93    |
| FMC*SS       | -16.68   | 0.01     | 0.00    |
| FMC*BT       | -16.65   | 0.01     | 0.00    |
| SS*BT        | -12.80   | 8.89     | 0.01    |
| FMC*SS*BT    | 21.10    | 3.83     | 0.000   |
| FC*FMC*SS*BT | -0.90    | 3.92     | 0.16    |
| ANOVA        |          |          |         |
|              | DF       | SS       | MS      |
| REGRESSION   | 15       | 33.47855 | 2.23190 |
| RESIDUAL     | 20       | 6.15489  | 0.30774 |
| F 7.25246    | SIGN.    |          |         |
|              | F.0.0000 |          |         |

DF=Degree of freedom FC= Feed composition FMC=Feed moisture content  
 SS= Sums of Square S\*S= Screw speed  
 MS=mean square BT= Barrel temperature

soybean addition in the feed stock affected the proximate composition and encouraged browning at elevated temperature and screw speed resulting in extrudates with decreases in solubility and reduced adsorptive capacities. Minimal experimental and predicted EMC were 7.50(g H<sub>2</sub>O/100g) and 7.28(g H<sub>2</sub>O/100g) respectively obtained at 25% FC, 35%FMC, 150rpm and 150°C while maximum experimental and predicted EMC were 11.50 and 11.56 respectively (Appendix I) obtained at 12.5%FC, 30%FMC, 180rpm and 175°C.

The estimated regression coefficient and ANOVA of the EMC of extrudates at 75% humidity is shown in Table 6.

The results showed that the process variables exhibited significant (P≤0.05) linear, cross product and quadratic effects. Analysis of variance showed that there was a significant (P≤ 0.01) model difference indicating that the model adequately fitted the linear regression. With coefficient of determination at 0.73 the model possessed predictive power showing about 73% of the variability within the model. Removing the non significant terms from the model the resulting polynomial became **water activity at 75% humidity = -81.71 -3.34FMC<sup>2</sup>TP -12.96FMC\*TP -12.26SS\*TP -1.88SS<sup>2</sup> -2.19TP<sup>2</sup>-----Eq4**

The response surface relationship (Fig. 7) between the EMC, SS and FMC showed that increasing the FMC and SS resulted in initial increases in extrudate EMC which later dropped from (10.90 to 7.05g H<sub>2</sub>O/100g). As the FMC and SS increased, extrudates with enhanced expansion exited the extruder with increased affinity for water. This occurred up to 20% FMC. Beyond this point, shear lowering and decreased BT resulted in increased residence time of extrudate. Extrudates high in bulk density and less active sorptive sites emerged leading to reduced adsorption capacity. Auta (2002) found that at aw of 0.6 - 0.75 condensed water is trapped, least strongly bound in the most mobile phase. It also constitutes the bulk phase water of any food product. This water is also trapped within the cells of food material. This implies that once the sorptive sites are disrupted, adsorption decreases. This is considered an attribute for the lowering of EMC with increased FMC.

The response surface plot of EMC, BT and FMC is shown in figure8. The figure showed that EMC increased with increased in BT, and FMC. Emerging extrudates therefore had greater expansion and hence less bulky with more sorptive sites exposed and greater gelatinized starch that made water adsorption easier. This was expected as high barrel temperature favoured high extrudate expansion resulting in increased adsorptive capacity.

However, further increases in the BT and FMC beyond 180°C and 30% resulted in reduced EMC. This was a typical food sorption response to increased temperature of processing or storage especially for a type II isotherm BET classification (Labuza 1984).

According to Stencl (1999) an increase in temperature causes a decrease in the amount of adsorbed water. This might be due to the fact that extrudates at higher temperature beyond 180°C and 30% FMC emerged less hygroscopic. In addition, Auta, (2002) reported that sorptive capacity of a product is reduced by pre-treatment or denaturation processes like heating, desalting and pH change. The results from this study agreed with this result. Minimum experimental and predicted values at this water activity were 7.80 and 7.33 respectively and were obtainable at 25%FC, 35%FMC, 150rpm and 150°C. Maximum experimental and predicted EMC values (12.46 and 12.40 gH<sub>2</sub>O/100g respectively), at this water activity were obtained at 37.5%FC, 20% FMC, 180rpm and 125°C.

The result of extrudate sorption isotherm at 90% relative humidity is shown in Table7.

The results showed that the process variables exerted significant (P≤0.01) linear, cross product and quadratic effects on extrudate equilibrium moisture content. The quadratic effect of FMC however exerted the most significant (P≤0.01) effect on the EMC of extrudate. Analysis of variance showed that there was a high significant (P≤0.01) model difference, indicating that the model fitted the linear regression. With coefficient of determination (R<sup>2</sup> = 0.84), the result showed that the model possessed high predictive power with about 84% of the total variation within the model. Removing the non-significant terms, the model equation for the EMC moisture at (0.9aw) became

$$\text{EMC (0.9aw)} = -110.89 -3.70M^2 +16.56 \text{ FMC} +23.88\text{FMCSS*TP} -19.50 \text{ FMCSS} -18.12\text{FMC*TP} -15.21 \text{ S*TP} +11.45 \text{ SS} +9.81 \text{ TP} \text{----- Eq5}$$

The results indicated that the quadratic influence of FMC was greater at 90% than at 64 and 75% humidities. The response surface relationship (Fig 9) between the EMC, FMC and SS showed that initial increases of moisture up to 20% and SS above 140 RPM resulted in increased EMC(Appendix I). Above these ranges however, EMC decreased due to the quadratic effect of FMC that was highly significant (P≤0.01). A similar trend was observed for the response surface plot of EMC, FMC and BT (Fig. 10). However, Fig 10 showed that increased BT and SS would result to lowering of EMC up to (9.0 g H<sub>2</sub>O/100g) if BT and SS exceeds 160°C and 140 rpm respectively. A similar response was obtained when the response surface plot of extrudate EMC was made against extruder BT and SS. The minimal experimental and predicted values of EMC at 0.90 aw were 7.99and7.55 (g H<sub>2</sub>O/100g) respectively obtained at 25%FC, 35%FMC, and 150rpm SS and 150°C BT.

Highest values of EMC for experimental and predicted data were 16.00and 16.01(g H<sub>2</sub>O/100g) respectively, obtained at 37.5% FC, 30% FMC, 180rpm SS and 175°C BT (Appendix I).

## MONOLAYER

The effects of processing variables on extrudate monolayer values are shown in Table 8. The results showed that extrudate monolayer values were not significantly ( $P \geq 0.05$ ) dependent on the independent process variables. Analysis of

variance showed that there was no model significance ( $P \geq 0.05$ ). With average coefficient of determination  $R^2 = 0.50$ , the model was not used for predictive purposes. However, the results were similar to those reported for snacks and crackers (Sunday, 2002).

**TABLE 6: Estimated Regression Coefficients and ANOVA For EMC At 0.75aw**

| Regression On    | Coefficients | Standard Error | P-VALUES | R <sup>2</sup> |
|------------------|--------------|----------------|----------|----------------|
| <b>Constants</b> | -81.87       |                |          |                |
| FMC              | 12.96        | 1.54           | 0.02     | .73            |
| SS               | 11.78        | 0.25           | 0.03     |                |
| BT               | 9.24         | 0.26           | 0.04     |                |
| FC*FC            | 0.42         | 2.91           | 0.50     |                |
| FMC*FMC          | -3.34        | 0.01           | 0.01     |                |
| SS*SS            | -1.88        | 1.85           | 0.10     |                |
| BT*BT            | -2.19        | 2.67           | 0.11     |                |
| FC*FMC           | 0.02         | 1.42           | 0.98     |                |
| FC*SS            | 0.21         | 2.37           | 0.79     |                |
| FC*BT            | -0.44        | 2.37           | 0.58     |                |
| FMC*SS           | -15.90       | 0.01           | 0.02     |                |
| FMC*BT           | -12.96       | 0.01           | 0.05     |                |
| SS*BT            | -12.96       | 0.00           | 0.05     |                |
| FMC*SS*BT        | 18.26        | 6.14           | 0.01     |                |
| FC*FMC*SS*BT     | -0.53        | 6.28           | 0.53     |                |
| ANOVA            | DF           | SS             | MS       |                |
| REGRESSION       | 15           | 41.91308       | 2.79421  |                |
| RESIDUAL         | 20           | 15.82112       | 0.79106  |                |
| F 3.53225        | SIGN.        |                |          |                |
|                  | F.0.00       |                |          |                |

DF=Degree of freedom FC= Feed composition FMC= Feed moisture content

SS= Sums of Square  
MS=mean square

S\*S= Screw speed  
BT= Barrel temperature

## CONCLUSION

The results obtained from this work showed that minimum values of EMC were obtained at higher levels of feed moisture content while significant increases in EMC were observed at lower levels of feed moisture content for all water activity levels evaluated. The results therefore established a trend showing that feed moisture content exercised the greatest influence on extrudate water sorption behaviour. The highest values of EMC were obtained at 30%FMC and 180rpm SS at all water activity levels analysed. This indicated that the effect of FMC and SS were more pronounced than barrel

temperature while feed composition did not exert significant ( $P \geq 0.05$ ) influence on extrudate EMC at all water activity levels evaluated. The results showed that predicted EMC values approximated experimental values. It was therefore concluded that the fitted polynomials were adequate in predicting the EMC of extruded acha and soybean blends. Though there was no model significance for extrudate monolayer values, the experimental values showed that the extrudates would remain chemically and microbially stable at ambient temperature storage.

**TABLE 7: Estimated Regression Coefficients and ANOVA for EMC at 0.90aw.**

| Regression On    | Coefficients | Standard Error | P-VALUES | R <sup>2</sup> |
|------------------|--------------|----------------|----------|----------------|
| <b>Constants</b> | -110.89      |                |          |                |
| FMC              | 16.56        | 1.49           | 0.00     | .84            |
| SS               | 11.81        | 0.24           | 0.01     |                |
| BT               | 9.81         | 0.25           | 0.01     |                |
| FC*FC            | 0.10         | 2.81           | 0.83     |                |
| FMC*FMC          | -3.70        | 0.01           | 0.00     |                |
| SS*SS            | 0.15         | 1.79           | 0.86     |                |
| BT*BT            | 0.15         | 2.58           | 0.88     |                |
| FC*FMC           | 0.25         | 1.37           | 0.68     |                |
| FC*SS            | 0.31         | 2.29           | 0.61     |                |
| FC*BT            | -0.07        | 2.29           | 0.91     |                |
| FMC*SS           | -19.50       | 0.01           | 0.00     |                |
| FMC*BT           | -18.12       | 0.01           | 0.00     |                |
| SS*BT            | -15.21       | 0.00           | 0.00     |                |
| FMC*SS*BT        | 23.88        | 5.93           | 0.00     |                |
| FC*FMC*SS*BT     | -0.94        | 6.07           | 0.16     |                |
| ANOVA            | DF           | SS             | MS       |                |
| REGRESSION       | 15           | 76.73238       | 5.10883  |                |
| RESIDUAL         | 20           | 14.77008       | 0.73850  |                |
| F 6.91780        | SIGN.        |                |          |                |
|                  | F.0.000      |                |          |                |

DF=Degree of freedom FC= Feed composition FMC= Feed moisture content

SS= Sums of Square  
MS=mean square

S\*S= Screw speed  
BT= Barrel temperature



TABLE 8: Estimated Regression Coefficients and ANOVA for Extrudate Monolayer values.

| Regression   | Coefficients | Standard Error | P-VALUES* | R <sup>2</sup> |
|--------------|--------------|----------------|-----------|----------------|
| On Constants | -12.50       |                |           |                |
| MC           | 4.62         | 2.40           | 0.51      | .50            |
| SS           | -0.23        | 0.39           | 0.97      |                |
| BT           | -0.61        | 0.41           | 0.92      |                |
| FC*FC        | -0.52        | 4.55           | 0.54      |                |
| MC*MC        | -0.96        | 0.01           | 0.53      |                |
| SS*SS        | -1.24        | 2.89           | 0.42      |                |
| BT*BT        | -1.08        | 4.17           | 0.56      |                |
| FC*MC        | -1.68        | 2.22           | 0.14      |                |
| FC*SS        | 0.65         | 3.71           | 0.56      |                |
| FC*BT        | 0.68         | 3.71           | 0.54      |                |
| MC*SS        | -2.58        | 0.01           | 0.77      |                |
| MC*BT        | -1.76        | 0.02           | 0.84      |                |
| SS*TP        | 4.08         | 0.00           | 0.62      |                |
| MC*SS*BT     | -4.08        | 9.60           | 0.97      |                |
| FC*MC*SS*BT  | 0.82         | 9.82           | 0.48      |                |
| ANOVA        |              |                |           |                |
|              | DF           | SS             | MS        |                |
| REGRESSION   | 15           | 34.25256       | 2.28350   |                |
| RESIDUAL     | 20           | 38.66920       | 1.93346   |                |
| F 1.18105    | SIGN.        | F              |           |                |
|              | 0.36         |                |           |                |

DF=Degree of freedom FC= Feed composition FMC= Feed moisture content  
 SS= Sums of Square S\*S= Screw speed  
 MS=mean square BT= Barrel temperature

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#### APPENDIX I: EXPERIMENTAL AND PREDICTED EMC VALUES USING THE MODELS DEVELOPED

| Extrusion runs | 0.35aw    |            | 0.40aw    |            | 0.64      |            | 0.75 aw   |            | 0.90aw    |            | Monolayer |            |
|----------------|-----------|------------|-----------|------------|-----------|------------|-----------|------------|-----------|------------|-----------|------------|
|                | Exp Value | Pred Value | Exp Value | Pred Value | Exp Value | Pred Value | Exp Value | Pred Value | Exp Value | Pred Value | Exp Value | Pred Value |
| 1              | 10.06     | 10.40      | 10.74     | 10.96      | 11.25     | 11.17      | 11.94     | 11.81      | 12.28     | 12.56      | 6.50      | ND         |
| 2              | 9.35      | 9.79       | 9.82      | 10.00      | 10.81     | 10.83      | 11.71     | 11.87      | 13.30     | 13.77      | 6.08      | ND         |
| 3              | 10.30     | 9.19       | 10.80     | 10.70      | 10.49     | 10.87      | 11.21     | 11.40      | 12.72     | 13.00      | 6.75      | ND         |
| 4              | 9.13      | 9.33       | 10.53     | 10.71      | 10.84     | 10.82      | 11.60     | 11.81      | 14.28     | 14.14      | 5.93      | ND         |
| 5              | 9.01      | 8.78       | 9.67      | 10.00      | 9.69      | 9.95       | 10.62     | 10.89      | 12.71     | 12.15      | 5.86      | ND         |
| 6              | 10.04     | 9.14       | 10.13     | 9.00       | 8.84      | 8.30       | 11.12     | 10.10      | 11.98     | 11.00      | 6.53      | ND         |
| 7              | 9.11      | 8.79       | 10.23     | 9.10       | 9.41      | 8.40       | 10.09     | 9.00       | 10.91     | 10.00      | 6.71      | ND         |
| 8              | 10.35     | 10.60      | 10.33     | 10.90      | 11.50     | 11.57      | 11.71     | 12.13      | 14.88     | 16.01      | 5.98      | ND         |
| 9              | 8.33      | 8.90       | 10.14     | 10.56      | 11.27     | 11.00      | 11.76     | 12.00      | 12.77     | 12.97      | 6.73      | ND         |
| 10             | 9.78      | 9.38       | 10.35     | 10.06      | 10.52     | 10.74      | 11.58     | 11.35      | 13.96     | 13.44      | 5.41      | ND         |
| 11             | 7.96      | 8.90       | 10.38     | 10.36      | 10.83     | 10.87      | 12.00     | 12.46      | 13.67     | 13.00      | 6.36      | ND         |
| 12             | 9.17      | 9.07       | 10.74     | 10.53      | 10.57     | 10.54      | 11.71     | 11.15      | 13.75     | 13.50      | 6.17      | ND         |
| 13             | 6.92      | 7.61       | 9.56      | 9.50       | 10.15     | 9.99       | 10.43     | 10.50      | 10.75     | 11.00      | 5.96      | ND         |
| 14             | 6.92      | 7.99       | 6.75      | 8.00       | 7.94      | 8.11       | 7.97      | 9.35       | 8.64      | 9.00       | 4.50      | ND         |
| 15             | 6.92      | 7.75       | 6.94      | 8.40       | 7.00      | 8.22       | 7.05      | 8.35       | 8.05      | 9.35       | 4.50      | ND         |
| 16             | 9.62      | 9.59       | 10.75     | 10.36      | 10.91     | 10.93      | 11.47     | 11.16      | 14.83     | 14.83      | 6.25      | ND         |
| 17             | 10.28     | 10.52      | 9.50      | 10.00      | 10.02     | 10.63      | 11.31     | 11.48      | 11.46     | 12.20      | 6.02      | ND         |
| 18             | 10.14     | 9.48       | 10.76     | 9.93       | 10.08     | 10.73      | 11.30     | 11.58      | 12.96     | 12.84      | 6.67      | ND         |
| 19             | 10.18     | 10.00      | 10.31     | 10.36      | 10.71     | 10.85      | 10.90     | 11.36      | 11.50     | 11.71      | 6.60      | ND         |
| 20             | 6.00      | 5.59       | 7.15      | 7.00       | 7.50      | 7.28       | 7.80      | 7.33       | 7.99      | 7.51       | 3.94      | ND         |
| 21             | 8.59      | 8.26       | 10.00     | 9.75       | 10.00     | 10.66      | 10.69     | 10.44      | 12.90     | 12.43      | 5.53      | ND         |
| 22             | 9.17      | 9.00       | 10.17     | 10.03      | 9.82      | 9.81       | 9.62      | 10.06      | 12.57     | 12.78      | 5.96      | ND         |
| 23             | 9.50      | 9.00       | 10.38     | 10.03      | 10.98     | 10.65      | 10.67     | 10.69      | 12.22     | 11.77      | 6.18      | ND         |
| 24             | 9.91      | 9.91       | 10.75     | 10.69      | 9.78      | 9.81       | 9.90      | 9.67       | 12.82     | 13.00      | 6.44      | ND         |
| 25             | 9.12      | 9.69       | 9.73      | 9.94       | 10.98     | 10.66      | 11.40     | 11.41      | 12.47     | 12.43      | 6.00      | ND         |
| 26             | 9.12      | 9.69       | 10.00     | 9.94       | 10.66     | 10.66      | 11.25     | 11.41      | 12.47     | 12.49      | 8.09      | ND         |
| 27             | 10.00     | 9.69       | 10.00     | 9.94       | 10.57     | 10.66      | 11.40     | 11.41      | 12.47     | 12.49      | 6.85      | ND         |
| 28             | 9.68      | 9.69       | 9.80      | 9.94       | 10.58     | 10.66      | 11.01     | 11.41      | 12.52     | 12.49      | 6.29      | ND         |
| 29             | 9.36      | 9.69       | 9.60      | 9.94       | 10.73     | 10.66      | 11.70     | 11.41      | 12.36     | 12.49      | 6.08      | ND         |
| 30             | 9.81      | 9.69       | 9.27      | 9.94       | 10.27     | 10.66      | 11.00     | 11.41      | 12.22     | 12.49      | 6.38      | ND         |
| 31             | 9.47      | 9.69       | 10.00     | 9.94       | 10.31     | 10.66      | 11.60     | 11.41      | 12.59     | 12.49      | 6.16      | ND         |
| 32             | 9.25      | 9.69       | 10.00     | 9.94       | 10.31     | 10.66      | 11.60     | 11.41      | 12.62     | 12.49      | 6.01      | ND         |
| 33             | 9.43      | 9.69       | 10.00     | 9.94       | 10.98     | 10.66      | 11.70     | 11.41      | 12.42     | 12.49      | 6.13      | ND         |
| 34             | 9.43      | 9.69       | 9.78      | 9.94       | 10.03     | 10.66      | 11.40     | 11.41      | 12.24     | 12.49      | 6.00      | ND         |
| 35             | 9.41      | 9.69       | 10.00     | 9.94       | 10.98     | 10.66      | 11.07     | 11.41      | 12.47     | 12.49      | 6.18      | ND         |
| 36             | 9.38      | 9.69       | 9.41      | 9.94       | 10.53     | 10.66      | 11.01     | 11.41      | 12.36     | 12.49      | 6.08      | ND         |

ND = Not determined because of low coefficient of determination  $R^2 < 0.60$  and lack of model significance ( $P < 0.05$ )

Exp value = Experimental value  
Pred value = Predicted value