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MOISTURE ADSORPTION ISOTHERMS AND ADSORPTION ISOSTERIC HEAT OF DRY GROUND MEAT

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Aktaş, N. (2022). Kuru kıymanın nem adsorpsiyon izotermleri ve adsorpsiyon izosterik ısısı. *GIDA* (2022) 47 (5) 717-728 doi: 10.15237/gida.GD22043

ABSTRACT
Dry ground meat is a cooked meat product. It is a popular meat product in Central Anatolian Region of Turkey. Calf plate or flank meats and intermuscular fats are used in the production of dry ground meat. In this study, sorption isotherms were determined using nine different salt solutions (0.1-0.9 a_w) having different relative humidity values at 5, 15 and 25°C. From the experimental data obtained, it was found that the sorption isotherms had Type-II characteristics. The obtained experimental data were applied to Iglesias-Chirife, Oswin, BET, Harkins-Jura, Smith, Henderson, Halsey, GAB, Peleg. Iglesias-Chirife and Peleg equations were revealed the best fitting. Isosteric heats of adsorption were evaluated by applying the Clausius-Clapeyron equation to experimental isotherms and decreased with increasing moisture content.
Keywords: Dry ground meat, sorption isotherm, isosteric heat

KURU KIYMANIN NEM ADSORPSİYON İZOTERMLERİ VE ADSORPSİYON İZOSTERİK ISISI

ÖZ
Kuru kıyma, pişirilmiş bir et ürünüdür. Türkiye'nin İç Anadolu Bölgesi'nde popüler bir et ürünüdür. Kuru kıyma üretiminde dana kaburga, karın etleri ve kaslararası yağ kullanılmaktadır. Bu çalışmada, farklı nispi nem değerlerine sahip dokuz farklı tuz çözeltisi kullanılarak sorpsiyon izotermleri 5, 15 ve 25°C'de belirlenmiştir. Elde edilen deneysel veriler Iglesias-Chirife, Oswin, BET, Harkins-Jura, Smith, Henderson, Halsey, GAB, Peleg eşitliklerine uygulanmış, en iyi uyumun Iglesias-Chirife ve Peleg eşitliklerinde olduğu ortaya çıkarılmıştır. Elde edilen deneysel verilerden sorpsiyon izotermlerinin Tip II özelliğe sahip olduğu bulunmuştur. Clausius Clapeyron eşitliği deneysel izotermlere uygulanarak izosterik adsorpsiyon ısıları belirlenmiş ve artan nem içeriği ile azaldığı tespit edilmiştir.
Anahtar kelimeler: Kuru kıyma, sorpsiyon izotermi, izosterik ısı

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INTRODUCTION

Dry ground meat is a traditional meat product produced by frying of the ground meat and fat in open cauldrons with the addition of salt. The block diagram of dry ground meat production is shown in Figure 1. In the production of dry ground meat, firstly, meat and intermuscular meat fat are ground. For this purpose, the ground meat and the ground meat fat are placed in the frying cauldrons and fried at 60°C and then 90°C for 30 minutes. Towards the end of the frying process, the salt is added and the frying process is continued for a while. After the frying process, melted animal fat is drained from the cauldrons, and it is kept for a certain period in a draining tank to separate the water it contains. The melted animal fat in the draining tank is poured over the ground meat taken into the molds, and then the cooling process is carried out and the product is offered for sale. The sustainability of traditional products like this, which are produced with intensive labor and reflect the cultural heritage of the society, has gained even more importance in the globalizing world. Therefore, the traditional food sector has to weigh improvements in the safety, health, and convenience of their products.

Ensuring food safety during the production and storage of such products is possible by revealing the characteristic properties of the products. Moisture sorption properties and the conditions associated with these properties are of great importance in this respect.

Water activity is one of the most important factors affecting the transfer of moisture in unit operations such as drying, cooling, freezing, heating, cooking and storage in foods. In addition, the stability of foods is closely related to water activity. Water activity varies depending on temperature, composition and physical state of food. At a given temperature and / or pressure, the relationship between the food's equilibrium moisture content and water activity is called sorption isotherm. These isotherms provide important information in characterizing of sorption. This information is important in modeling the drying process, designing and optimizing drying equipment, determining shelf life, calculating changes in moisture content during storage, and selecting the most suitable packaging material for food (Shi et al., 2016; Trujillo et al., 2003).

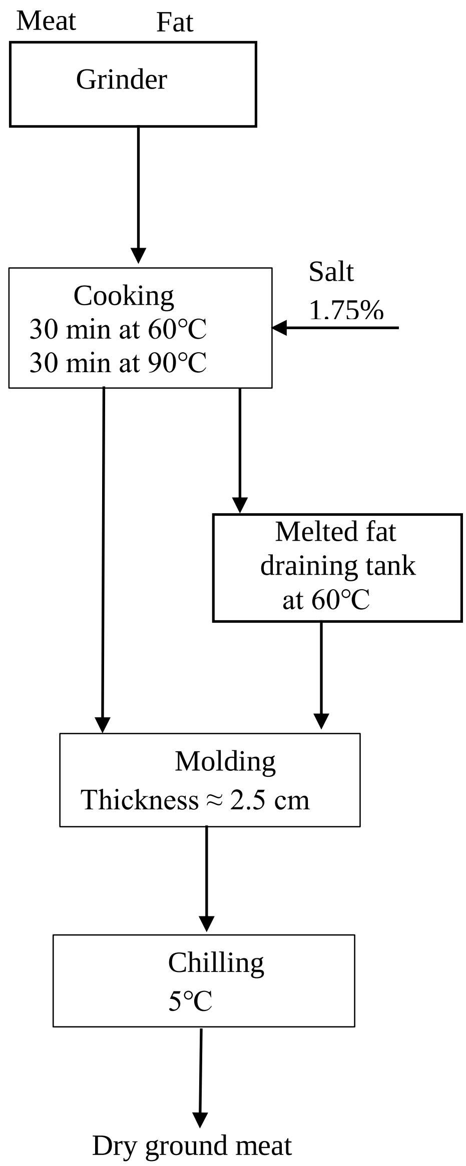

Figure 1. Block diagram of dry ground meat production.

Knowledge of the heat of sorption in food processes such as drying and cooking is extremely important to control the heat and mass transfer processes and moisture changes during storage. The heat of sorption indicates the magnitude of the bonding energy or the intermolecular interaction forces between the adsorbent surface and the adsorbate molecules (Chung and Pfost, 1967). The high values of isosteric heat indicate that the bond energy between the adsorbent surface and the water vapor molecules is low or the intermolecular forces are weak, and if it is small, these forces and bond energies are strong (Basu et al., 2006; Al-Muhtaseb et al., 2002; Mrad et al., 2012; Mbarek and Mihoubi, 2019). There has been no research report conducted on determining the moisture sorption isotherms of dry ground meat. Therefore, the objectives of this study were 1) to determine the water sorption isotherms for dry ground meat at different temperatures (5, 15 and 25°C), 2) to find mathematical models that provide an accurate prediction of the isotherms, 3) to determine the net isosteric heat of sorption.

MATERIALS AND METHODS

Materials

Dry ground meat samples with triplicate, which were produced on separate processing days by the same production technique, were purchased from a local company (Kavdırlar, Nevşehir, Turkey).

Methods

For each dry ground meat, pH, moisture, fat, protein and ash were determined according to Ockerman (1985) with three replicates. Thiobarbituric acid reactive substances (TBARS) analysis in dry ground meats were determined based on Lemon (1975).

Adsorption isotherms

An isopiestic method was used to determine the adsorption isotherms as previously described (Gal, 1975). Briefly, adsorption isotherms were measured with desiccators containing various saturated salt solutions that provide a_w value of between 0.1 and 0.9 (Figure 2). The temperatures used for the adsorption isotherms were 5, 15 and 25°C. Samples were first dried to level containing 3.85±0.11% water contents for sorption in air-dryer at 35°C and they were weighed into Quickfit® glass bottles of 50 mg parts with 0.1 mg sensitivity. Then, they were kept half-open to avoid any confusion in the caps of the bottles in desiccators containing various salt solutions providing the desired relative humidity for 5 days to "equilibrate" until there was no noticeable weight change in triplicate experiment, as evidenced by constant weight values (± 0.01 mg). In order to prevent the microbial spoilage of the samples, crystalline thymol was placed in the desiccators. Thereafter, the bottles were taken from the desiccator and the weight gain was measured (Bel M21 4Ai, Bel Engineering, Italy). The water content of each sample was determined by drying at 105±1°C until they reach to a constant weight. Water content was expressed as kg water/kg dry matter. The saturated salt solutions used were prepared a week beforehand and allowed to gain stability by mixing every day. The following saturated salt solutions and relative humidity media were used: LiCl 11.30%, CH_3COOK 23.11%, $MgCl_2$ 33.07%, K_2CO_3 43.16%, $Mg(NO_3)_2$ 54.38%, $NaNO_2$ 65.40%, NaCl 75.47%, KCl 85.11% and $BaCl_2$ 90.69% (Bell & Labuza, 2000). The relative humidities are values at 20°C. The equilibrium relative humidity values in the ambient inside the desiccators were measured by electronic relative humidity meter. All measurements were made on triplicate samples.

Fitting of adsorption data to various isotherm equations

Although there are several mathematical models available to depict the water sorption isotherms of foods, none of them are able to give accurate results for the whole range of water activities, and for all types of foods. Moreover, it is almost impossible to find a one model that fits all of the cases due to the complexity of food components; instead, various models have been proposed and checked for fit to experimental data. Thus, in this study, the experimental data obtained were applied to different isotherm equations that are as follows: Iglesias-Chirife, Oswin, BET, Harkins-Jura, Smith, Henderson, Halsey, GAB, Peleg, modified Chung-Pfost, modified Oswin. The linearized forms of the Iglesias-Chirife, Oswin, BET, Harkins-Jura, Smith, Henderson, Halsey relationships were used for calculating the parameters of the isotherm equations using a linear regression programme (SPSS 22 for Windows). The parameters of the GAB, Peleg, modified Chung-Pfost, modified Oswin models were estimated from the experimental results using the nonlinear regression analysis (SPSS 22 for Windows).

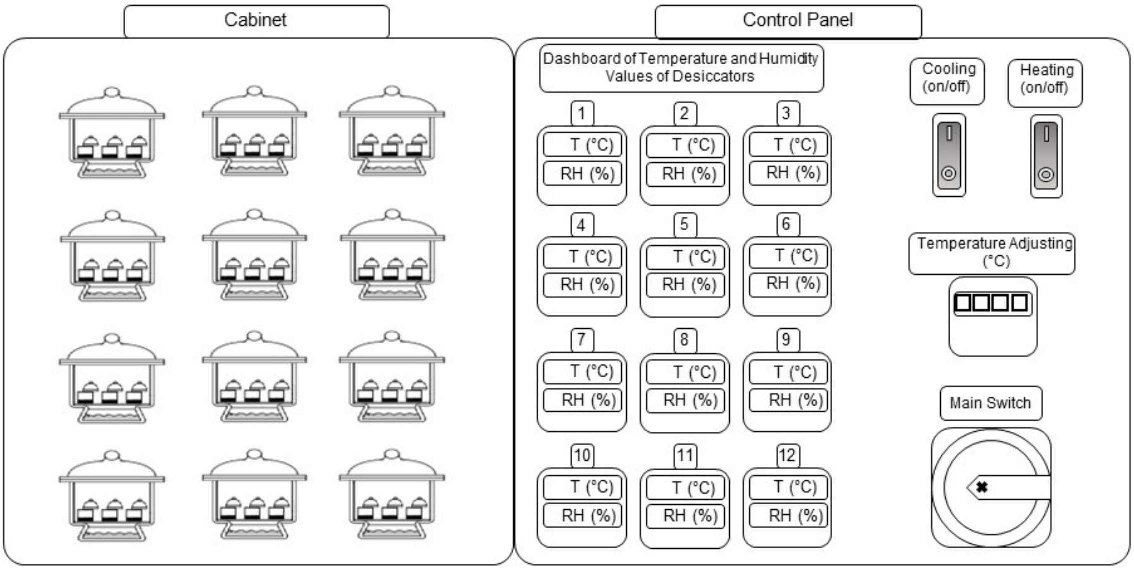

Figure 2. Experimental apparatus for determination of the adsorption isotherms

The goodness of fit as applied to the experimental data were evaluated through the determination coefficient (R^2), mean relative percentage deviation (P) and standard error of estimate (SEE).

$$R^2 = 1 - \frac{\text{Residual sum of squares}}{\text{Corrected sum of squares}} \quad (1)$$

$$P = \frac{100}{n}\sum_{i=1}^{n}\frac{|X_i - X_{pi}|}{X_i} \quad (2)$$

$$SEE = \sqrt{\frac{\sum(x_i - x_{pi})^2}{d_f}} \quad (3)$$

where X_{pi}, predicted, X_i, experimental moisture contents and d_f, the number of degree of freedom (number of data points minus number of constants in the model). It was reported in several previous studies that the R^2 and SEE values may not be sufficient evidence fort the goodness-of-fit (Chen and Morrey, 1989; Sun, 1999; Aviara et al., 2004; Iguaz and Virseda, 2007). For this reason, the mean relative percentage deviation (P) gives an idea of the mean departure of the measured data from the predicted data and is used to describe the goodness-of-fit of an equation. Therefore, the smaller the P value, the better the goodness-of-fit. A model is considered acceptable if the P values are below 10% (Ansari, Farahnaky, Majzoobi, & Badii, 2011; Mrad, Bonazzi, Boudhrioua, Kechaou, & Courtois, 2012).

Isosteric heat of adsorption

The isosteric heats of adsorption were calculated by using Eq. (4), which originates from the Clausius–Clapeyron equation, to the adsorption isotherms at different temperatures (Tsami, 1991).

$$\frac{d \ln a_w}{d\left[\frac{1}{T}\right]} = -\frac{Q_n^{st}}{R} \quad (4)$$

Plotting the adsorption isotherms as ln aw versus 1/T for certain values of moisture content, the net sorption isosteric heat Q_n^{st} can be obtained from the slope of these representations. In order to be able to use this method, sorption isotherms

at more than two temperatures are required as the simplifications for Eq. (4) were made.
The isosteric heat of sorption (Q^{st}) is calculated by including the latent heat of vaporization for pure water (L_r) to the net isosteric heat of sorption (Eq. (5)), considering L_r as the average value for the temperatures taken into account (2466.18 J/g for 5–25 °C).

$$Q^{st} = Q_n^{st} + L_r \quad (5)$$

RESULTS AND DISCUSSION

Physicochemical analysis

The results of physicochemical analysis were shown in Table 1. pH values of the dry ground meats were higher than that of the raw meat (pH= 5.64±0.02). During the cooking, the pH of the meat increases. The increase in the pH values might be occurred due to exposure to imidazolium during cooking, the basic R group of the amino acid histidine. Moisture content of the dry ground meat samples was 51.82%. This value is higher than the moisture content of kavurma (45%) (Turkish Food Codex, 2019). This situation might be significantly affect the storage period at 4°C. However, dry ground meat is not a product that could be stored for a very long time like kavurma. Generally, productions are made for short-term purposes (15 days) and offered for sale.

Table 1. Physicochemical characteristics of dry ground meat samples

pH	6.26±0.03
Moisture (%)	51.82±0.10
Protein (%)	24.62±0.18
Fat (%)	21.52±0.48
Ash (%)	2.04±0.22
TBARS (μmol MDA/ kg)	6.28±0.17

Cooking is a main process of dry ground meat production. The cooking process of meat and fat by grinding could be caused much more oxidation. TBARS value of dry ground meat is shown in Table 1. The occurrence of lipid oxidation is an undesirable situation as it causes changes in taste, aroma, odour, texture and color in meat and meat products and results in quality losses (Ockerman, 1985). A significant amount of animal fat is used in the production of dry ground meat. After the production, the preservation is carried out at 4°C and during this time the sale is made in open containers. This situation creates important problems in terms of lipid oxidation during storage and sale. In order to preserve the quality characteristics of the product, it must be packaged with suitable packaging material.

Adsorption isotherms

Adsorption isotherms of the dry ground meat samples determined at three different temperatures are given in Figure 3. The isotherms revealed a typical type-II shape according to the BET classification. This type is commonly seen in food materials containing high protein content and corresponds to multilayer formation (Figure 3). In the range of 11% to 43% relative humidity, it can be seen that the equilibrium moisture content was almost constant, resulting in an almost horizontal line. A similar situation was determined as a result of the research carried out in bologna by Igbeka and Blaisdel (1982). The researchers interpreted this situation as an increase in environmental relative humidity would only have a slight effect on the stability of the product.

It has been also reported that the amount of sorbed water in protein-rich foods mainly depends on the number and presence of two types of hydrophilic groups (polar side chains and carbonyl and imido groups of peptide bonds) that are capable of binding water through hydrogen bond formation (Singh et al., 2001; Aktaş and Gürses, 2005). Water sorption by proteins occurs on the polar side chains at low humidities, spreads out to peptide linkages and then leads to multilayer adsorption at high humidity.

The increase in temperature caused a reduction in the equilibrium moisture content (Figure 3). However, an increase in the temperature did not cause the formation of any crossover or intersection point in the curves. The differences of equilibrium moisture contents between three temperatures were found statistically significant

(P<0.05). This tendency formed by the increase in temperature could be ascribed to the excitation states of water molecules. In addition to an escalation in the kinetic energy of the molecules that also leads to increases of molecular mobility, a rise in the temperature causes a reduction in the attractive forces between molecules. Therefore, at low temperature values, water molecules having slow molecular mobility are bound more easily to convenient binding sites on surface and promotes an increase in the degree of water sorption by the product with decreasing temperature at the given water activity. (Quirijns et al., 2005; Ansari et al., 2011; Mbarga et al., 2017).

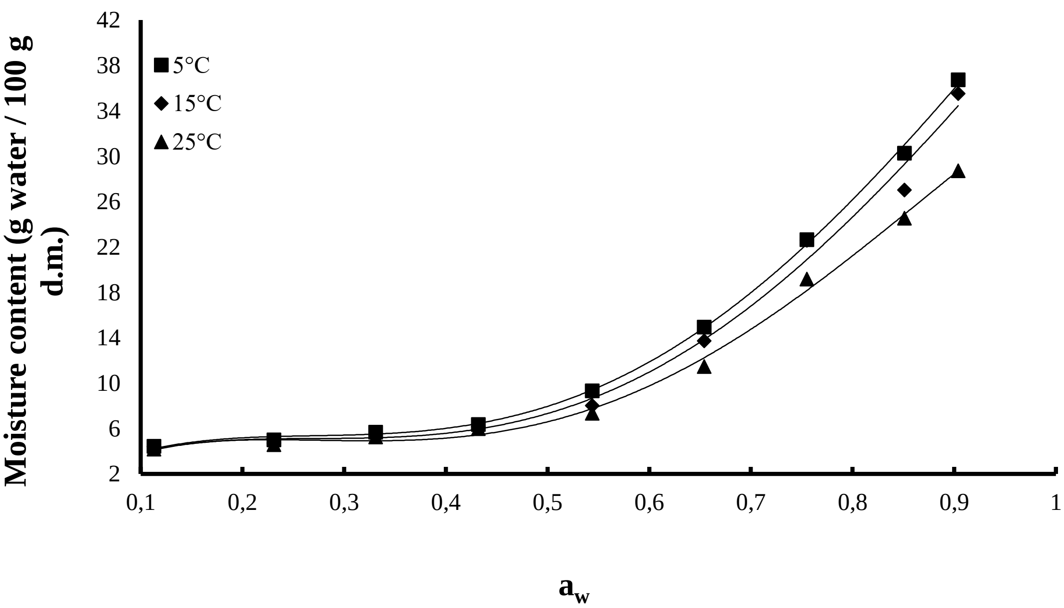

Figure 3. Moisture adsorption isotherms of dry ground meat samples at 5°C, 15°C and 25°C.

Fitting of adsorption data to various isotherm equations

The lowest P values for the isotherm equations at 5°C, 15°C and 25°C were obtained when Iglesias-Chirife equation was used (Figure 4), which was followed by Peleg. The highest P values were determined Modified Chung Pfost and Henderson equations, respectively (Tables 2). In this present study, Iglesias-Chirife and Peleg equations revealed the best fitting. Polatoğlu et al., (2011; 2013) reported that Peleg model gave the best fit for sucuk (Turkish dry-fermented sausage) and pastırma (Turkish dry meat product). Peleg's model parameters (A, B, C and D) showed a decrease with increasing temperature. The Peleg equation can predict both sigmoidal and non-sigmoidal isotherms. According to Peleg (1992), this model fitted as well as or better than the GAB model but its constants have no physical meaning.

Compared to the water activity range of 0.1-0.9 in terms of fit to the BET equation, the adsorption data obtained in the range of 0.1–0.55 water activity was found to fit better the BET model (Table 2). This agrees with report that the model of BET is the best fit for this water activity range (Labuza et al., 1985). The P values for the model of BET in the water activity range 0.1–0.55 were found to be 5.76, 5.29, 1.52 at 5, 15 and 25 °C, respectively. The high P values obtained in the 0.1-0.9 water activity range could be attributed to the existence of multiple mechanisms for sorption.

Table 2. Estimated parameters for several models for isotherms of dry ground meat samples at different temperatures

Isotherm equation	a_w (range)	Temperature (°C)	Constant parameters	Determination coefficient (R^2)	P (%)	SEE
Iglesias and Chirife $\ln\left(m+\left(m^2+m_{0.5}\right)^{\frac{1}{2}}\right)=p+b\left(a_w\right)$	0.1-0.9	5°C	b= 2.83 p= 1.60	0.951	0.16	0.008
		15°C	b= 2.77 p= 1.58	0.938	0.43	0.022
		25°C	b= 2.57 p= 1.60	0.939	0.29	0.021
Oswin $\ln m=\ln k+n\ln\left(\frac{a_w}{1-a_w}\right)$	0.1-0.9	5°C	n= 0.56 k= 10.12	0.939	15.44	0.638
		15°C	n= 0.55 k= 9.55	0.926	17.00	0.689
		25°C	n= 0.51 k= 8.72	0.929	15.78	0.604
BET $\frac{a_w}{m(1-a_w)}=\frac{1}{(m_0\,C)}+\frac{(C-1)}{(m_0\,C)}\,a_w$	0.1-0.9 /0.1-0.5	5°C	C=46.94/60.34 m_o= 4.35/4.04	0.866/0.973	12.28/5.76	0.815/0.206
		15°C	C= 44.62/166.41 m_o= 4.07/3.53	0.868/0.998	11.78/5.29	0.750/0.157
		25°C	C=35.44/-53.75 m_o= 3.24/3.32	0.869/0.999	12.33/1.52	0.717/0.041
Harkins-Jura $\frac{1}{m^2}=\left(\frac{B}{A}\right)-\left(\frac{1}{A}\right)\log a_w$	0.1-0.9	5°C	A= 16.34 B= -0.05	0.960	13.37	0.829
		15°C	A= 15.65 B= -0.03	0.941	14.44	0.940
		25°C	A= 14.53 B= -0.02	0.960	13.00	0.784
Smith $m=W_b-W\ln(1-a_w)$	0.1-0.9	5°C	W= 15.60 W_b= 0.28	0.978	19.11	0.538
		15°C	W= 14.74 W_b= 0.31	0.968	21.22	0.637
		25°C	W= 12.08 W_b= 0.49	0.969	18.00	0.500
Henderson $\ln[-\ln(1-a_w)]=\ln k+n\ln m$	0.1-0.9	5°C	n= 0.78 k= 14.96	0.880	23.33	1.121
		15°C	n= 0.77 k= 14.03	0.861	26.11	1.160
		25°C	n= 0.71 k= 12.47	0.864	23.55	0.996
Halsey $\ln m=\left[\frac{1}{n}\ln c\right]-\left(\frac{1}{n}\right)\ln\left[\ln\frac{1}{a_w}\right]$	0.1-0.9	5°C	n= 1.28 c= 11.87	0.969	12.11	0.657
		15°C	n= 1.30 c= 11.45	0.961	12.44	0.582
		25°C	n= 1.40 c= 12.65	0.964	11.22	0.527

Table 2. (Continued)

Isotherm equation	a_w (range)	Temperature (°C)	Constant parameters	Determination coefficient (R^2)	P (%)	SEE
GAB $\frac{a_w}{m} = \alpha a_w^2 + \beta a_w + \gamma$ $\left[\frac{-1}{(4 \alpha \gamma - \beta^2)}\right]^{\frac{1}{2}}$ $c = \left[\beta - \frac{\left(\frac{1}{m_m}\right)}{(-2 \gamma)}\right]$ $K = \frac{1}{m_m c \gamma}$	0.1-0.9	5°C	m_m=4.33 c=1.01 K'=28.23	0.850	12.60	1.067
		15°C	m_m=3.94 c=1.03 K'=41.07	0.856	14.58	1.260
		25°C	m_m=3.69 c=0.95 K'=92.02	0.891	7.13	0.414
Peleg $M = A a_w^B + C a_w^D$	0.1-0.9	5°C	A= 46.33 B= 3.37 C= 3.72 D= -0.10	0.998	4.49	0.176
		15°C	A= 44.36 B= 3.43 C= 3.38 D= -0.14	0.990	7.25	0.379
		25°C	A= 36.13 B= 3.34 C= 3.35 D= -0.12	0.993	6.19	0.254
Modified Chung-Pfost $M = -\frac{1}{c}\ln\left[\frac{(t + b)\ln(a_w)}{-a}\right]$	0.1-0.9	5°C	a= 78.76 b= 34.24 c= 0.09	0.931	28.22	1.088
		15°C	a= 34.87 b= 2.40 c= 0.92	0.918	31.67	1.089
		25°C	a= 557.54 b= 230.96 c= 0.11	0.921	20.11	0.657
Modified Oswin $(A + B t)\left(\frac{a_w}{1- a_w}\right)^C$	0.1-0.9	5°C	A= -3.86x10-4 B= 7.73x10^3 C= 0.59	0.978	14.22	0.591
		15°C	A= -7.74x10^4 B= 5.16x10^3 C= 0.60	0.971	15.01	0.537
		25°C	A= 1.73x10^4 B= -690.73 C= 0.56	0.966	14.89	0.569

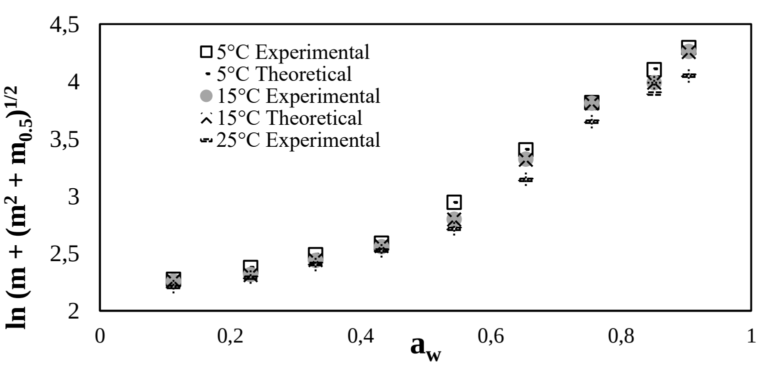

Figure 4. Moisture adsorption isotherms of the dry ground meat plotted by using Iglesias-Chirife model

The monolayer water contents (shown as m_o from the BET equation, and m_m from the GAB equation) are shown in Table 2. Above monolayer moisture content, which is assessed as a critical water content could result some deteriorations in foods such as activity of enzymes, non-enzymatic browning and lipid oxidation. However, below monolayer moisture contents where water is strictly adsorbed to the food, water is not able to serve as a solvent or substrate in any reaction except for lipid oxidation. When temperature increased, monolayer moisture content decreased (Table 2). The decrease in the content of monolayer moisture could result in a drop in the abundance of active sites because of the physical and chemical alterations caused by temperature. This agrees with previous reports (Falade and Aworh, 2004; Vega-Galvez et al., 2009; Ansari et al., 2011; Mrad et al., 2012; Prasantha and Amunogoda, 2012; Badii et al., 2014; Mbarga et al., 2017). m_o content in the BET isotherm equation could be an indicator for the affinity of polar sites to water vapour. Iglesias and Chirife (1976) suggests that there is a good correlation between the number of water molecules calculated to exist in a B.E.T. monolayer and the number of polar side chains. This suggests that each polar group initially sorbs one molecule of water followed at higher humidities by multimolecular adsorption. Moreover, lower m_o values were calculated when BET equation was used, compared to those calculated in the GAB at all temperatures tested. This finding is also in an agreement with the findings reported by other researchers (Timmermann et al., 2001; Mrad et al., 2012; Aykın and Erbaş, 2016; Aykın-Dinçer and Erbaş, 2018). These differences can be attributed that the model of BET mainly focuses on surface adsorption in the first layer, whereas the model of GAB also considers sorbed water properties in the region of multilayer (Bell and Labuza, 2000). In other words, the GAB model takes into account both capillary saturation and surface saturation (Aykın-Dinçer and Erbaş, 2016).

Isosteric heat of adsorption

The adsorption isosteric heat is shown in Figure 5. When the moisture content increased, a decrease in sorption energy was observed. This could be attributed to the fact that the adsorption at first takes place in the most active and accessible sites that involve elevated energies of interaction. Once these sites are engaged, adsorption to the less active sites having poorer interaction energies starts (Iglesis and Chirife, 1976; Sanchez et al., 1997). At low water content, binding seems to be predominantly controlled by Van der Waals forces and hydrogen bonding occuring between the adsorbent surface (dry ground meat) and water molecules.

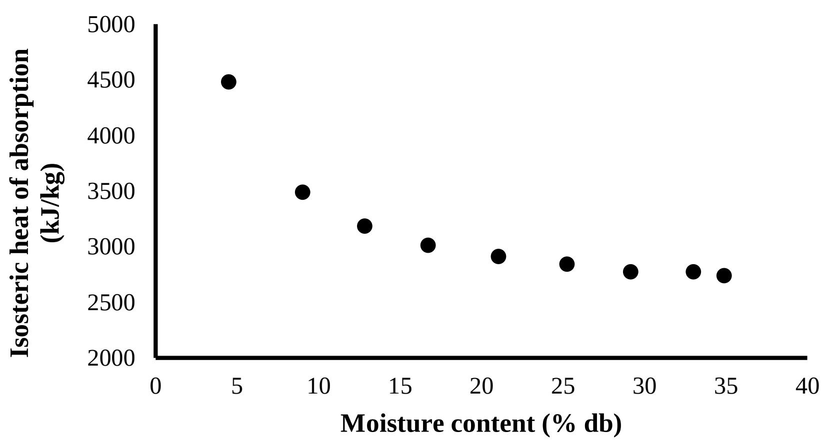

Figure 5. Isosteric heat of adsorption

CONCLUSION

The results obtained in this study clearly indicates that sorption isotherms of dry ground meat samples were type II isotherm pattern that is commonly observed in high protein content food products. The equilibrium moisture content relationship of the dry ground meat samples in the studied temperatures were best described by the Iglesias-Chirife and Peleg models.

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CONFLICTS OF INTEREST

Author declare that there is no conflict of interest. This article does not contain any studies with human or animal subjects.

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