Moisture sorption isotherm and shelf-life estimation of freeze-dried surimi powder

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Abstract. Due to the fact that commercial wet surimi must be frozen, the adoption of dried surimi or surimi powder has emerged as the favored choice in the surimi industry to reduce costs. However, as surimi experiences the transition to its dried form, changes in moisture content can potentially influence quality and shelf life. To determine the optimal storage conditions for surimi powder, moisture sorption isotherms were evaluated over a range of water activity from 0.069 to 0.970. Five sorption models were then applied to the data. The water vapor permeability of three different packaging materials was also determined and the Labuza mathematical model was used to predict the shelf life of the product. This study attempted to assess the changes in the quality of surimi powder and predict its shelf life. The moisture sorption isotherm curve for dried surimi revealed a smooth sigmoid pattern, signifying equilibrium moisture content. The chosen sorption isotherm model, the Hasley formula (Me = log(log(ln(1/aw)) + 1.893) /-2.209), generated the mean relative determination value of 2.31. The Labuza model estimated the shelf life of dried surimi, revealing a predicted shelf life of 22.6, 4.5, and 6.1 months with retort pouch, HDPE, and OPP packaging, respectively.

1 Introduction

Initially, surimi described the processed and preserved concentrate of myofibril protein derived from minced fish muscle tissues that have been rinsed with water [1]. Surimi is an important intermediate food product that is primarily utilized in the production of various seafood items and other analog surimi-based foods. In recent decades, the primary raw materials used for surimi production have been utilized marine-based species, including Alaska pollock, Threadfin bream, and Pacific whiting. The production of surimi commences with the utilization of white-fleshed fish sourced from cold waters. Meanwhile, tropical fish have gained popularity in this decade. Although, Alaska pollock still supplied 50% of the global surimi demand since 2000. Alaska pollock is regarded as a surimi basic material of the highest quality, producing 250,000 tons every year.

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However, this current decade, economically significant fish species such as Alaska pollock and Atka mackerel have encountered substantial declines, with the Alaska pollock population declining by around 50% and now being classified as critically endangered due to overfishing. The surimi industry has experienced tremendous expansion, although there is a shortage of fisheries resources [2]. Therefore, a decrease in surimi raw materials could have a big influence on the global demand for the surimi industry. Hence, it is crucial to utilize cost-effective resources derived from freshwater species in order to establish and maintain sustainable fisheries industries. The utilization of freshwater fish as the primary source for surimi production is expected to ensure a consistent supply of raw materials. Substituting catfish and tilapia, the second most widely cultivated fish following carp, for the surimi industry as raw material sources is feasible [3-7].

Commercial wet surimi requires storing at temperatures below freezing. Hence, the adoption of dried surimi or surimi powder has emerged as the favored choice in the food industry to reduce costs. By rehydrating surimi powder with four times its weight in water, it is possible to transform it into wet surimi; the water content of the rehydrated wet surimi powder would be comparable to that of a frozen surimi block. Additionally, surimi powder is advantageous because it can be stored at room temperature rather than being frozen and has a reduced cost of distribution. Furthermore, it is practical to handle and store, and it can be used effectively in dry mixtures [6, 7]. Additional fish species that have been studied as potential sources for surimi powder include capelin (*Mallotus villosus*) and thread bream (*Nemipterus* sp.) [8, 9], tilapia and fat sleeper (*Dormitator moculatus*) [10], lizardfish (*Saurida tumbil*)**,** Pacific whiting (*Merluccius productus*) [11-13], marlin (*Makaira* sp.)**,** carp (*Cyprinus carpio*), and saithe (*Pollachius virens*) [14,15].

Our previous findings [7] revealed that adding cryoprotectants, especially trehalose, effectively preserves the quality of dried surimi from catfish. We also succeeded in incorporating small proteins into tilapia to enhance the surimi gel [3]. Additionally, we reported on the role of cryoprotectants in keeping the physical properties of tilapia-dried surimi [6]. With numerous reports on surimi powder from various raw material sources and optimization of its production, there remains unclear information on the shelf life of surimi powder. However, surimi powder or freeze-dried surimi is a dry product that often experiences deterioration due to environmental changes. Dry products generally have a relatively long shelf life, but those with high porosity are sensitive to high humidity.

Numerous studies have explored the shelf life of surimi, however with a predominant focus on its evaluation in wet form or as wet surimi to monitor changes during frozen storage periods. Only one study has addressed the shelf-life evaluation of surimi powder. Nath and Sing [16] conducted an assessment on dry surimi powder made from *Pangasianodon hypophthalmus*, demonstrating its ability to maintain quality for up to 210 days under chilling condition (4 °C) without exceeding acceptable limits.

In fact, various techniques for shelf-life estimation have been established, including actual shelf life or extended shelf-life calculation (ESS), and accelerated shelf-life testing studies (ASS or ASLT). Conducting ESS shelf-life testing through the storage of a packaged product under actual storage settings incurs significant expenses and requires an extensive period of time. Consequently, there is a need for a cost-effective and expeditious shelf-life simulation model. Many studies have been conducted on the determination of food product shelf life utilizing accelerated models that account for food product deterioration in relation to storage temperature [17]. Furthermore, the aforementioned study [16] on the measurement of surimi powder was carried out under chilling condition, necessitating the use of high energy. The current state of uncertainty pertains to the potential impact on the surimi condition if the sample were to be stored at ambient temperature.

By utilizing a mathematical model based on the Arrhenius relationship and relative humidity, Cardoso and Labuza [18] were able to forecast the expiration life of food products.

The water activity (a_w) of food products at specific temperatures and pressures can be determined using the water sorption isotherm curve and the equilibrium moisture content. The composition of dried materials, their physical structure and temperature are all variables that have the potential to impact the isotherm sorption curve. This curve strongly correlates with the stability of food materials across a range of processing requirements and storage conditions, in addition to representing the a_w value within its composition.

The packaging of dry food products plays a critical function in preserving their shelf-life. Similar to Fikri [19], who modeled the moisture sorption isotherm of gelatin powder, this study also employs five models to assess the accuracy of the moisture sorption isotherm curve of tortilla products, including the Oswin, Chen-Clayton, Hasley, Caurie, and Henderson models. Hence, the aforementioned models, based on previous research, are capable of depicting the moisture sorption isotherm curve in dried products [17]. However, as far as the authors are aware, there are no published studies on the moisture sorption of freeze-dried surimi powder under various humidity and packaging conditions. Therefore, determining the shelf life of dry products, especially surimi powder, is influenced by several internal and external factors. Determining the shelf life of dry products, especially surimi powder, is crucial and should be estimated using critical moisture content models. The critical moisture content model is determined by variations in air humidity, and the moisture sorption model of food ingredients needs to be further studied to prepare this product for industry.

2 Methods

2.1 Materials

The raw material utilized was catfish surimi flour. The chemical substances used include Pro Analysis-grade K_2CO_3, NaI, K_2SO_4, $MgCl_2$, NaOH, KCl, NaCl, KI, $NaNO_2$, NaBr (Merck, for analysis), plastic packaging materials; OPP, HDPE, retort pouch (PET 12/Aluvo 7/LLDPE 40), vaseline, and distilled water. The equipment utilized comprises storage apparatus and analytical instruments. The storage apparatus for dried surimi includes a modified desiccator for controlling relative humidity (RH). The freeze dryer employed for surimi drying is the Christ Alpha 2-4 3360 Harz type 10042. Laboratory instruments include dissection tools and glassware, an a_w meter (Shibaura Electronics WA-360), a freezer (SHARP) type FRV-200, and a refrigerator (GR K262/262PD, Glacio-Toshiba).

2.2 Estimation of the shelf life of freeze-dried surimi

The production of surimi powder refered to our previous report [6]. Subsequently, the surimi powder was packaged using three types of packaging: retort pouch (PET 12/Aluvo 7/LLDPE 40), HDPE, and OPP. The shelf life of freeze-dried surimi was determined using the critical water content model. The next experiments were conducted with the aim of generating the water sorption isotherm curve for the freeze-dried surimi, which was used to predict shelf life. Various modeling approaches were employed to determine the quality degradation parameters of freeze-dried surimi powder. Observations include measurements of shelf-life prediction, critical moisture content, and equilibrium moisture content analysis based on the transport of water vapor into the packaging material and water absorption by the food material, utilizing mathematical models with regression analysis as follows:

$$t = \frac{\ln\left(\frac{Me - Mi}{Me - Mc}\right)}{\left(\frac{k}{x}\right)\left(\frac{A}{Ws}\right)\left(\frac{Po}{b}\right)} \tag{1}$$

qhere: t = shelf life or time to reach critical moisture content (days), Me = equilibrium moisture content of surimi (gH_2O/g solid), Mi = initial moisture content of surimi (gH_2O/g solid), Mc = critical moisture content of surimi (gH_2O/g solid), k/x = permeability constant of the packaging material to water vapor (g/m^2.day.mmHg), A = surface area of the packaging (m^2), Ws = weight of solids per packaging (g), Po = vapor pressure of water in the storage space (mmHg), b = slope of the moisture sorption isotherm curve.

2.3 The testing protocol for variables relating to the estimation of shelf life

The estimation of the shelf life of freeze-dried surimi using the critical moisture content approach model initiated with the identification of several variables essential for the shelf-life calculation. The testing procedure for these variables included the determination of initial moisture content, critical moisture content, equilibrium moisture content, the generation of water sorption isotherm curves, determination of the isothermic sorption equation model, model evaluation, determination of the slope value (b) of the isothermic sorption curve, as well as the determination of the weight of solids per packaging and the surface area of the packaging.

2.3.1 Determination of the initial moisture content (Mi)

The determination of the initial moisture content was crucial to understand the product's initial condition. The assessment of the initial moisture content in freeze-dried surimi was carried out on samples freshly opened from the packaging. The determination of the initial moisture content (Mi) followed the standardized method outlined in AOAC 2005 for moisture content determination

2.3.2 Determination of the critical moisture content (Mc)

The determination of the critical moisture content (Mc) started by storing the freeze-dried surimi product without packaging at room temperature (30±1 °C) for 5 hours. Samples were extracted every hour to assess water activity and were analyzed their moisture content. The moisture content of freeze-dried surimi was measured in accordance with AOAC (2005). The critical moisture content was derived from a linear regression equation linking water activity values to moisture content values, with the critical moisture content determined when the water activity value reached 0.80. The critical state of freeze-dried surimi was identified when the water activity value approached the final threshold criteria for dry food products.

2.3.3 The determination of equilibrium moisture content (Me)

The determination of equilibrium moisture content (Me) was initiated by dissolving specific salts until saturation or insolubility was reached. The salts used included K_2CO_3, NaI, K_2SO_4, $MgCl_2$, NaOH, KCl, NaCl, KI, $NaNO_2$, and NaBr. A volume of 100 ml of a saturated salt solution was introduced into a modified desiccator to control room relative humidity (modified desiccator). Approximately 3 g of freeze-dried surimi sample was placed in a pre-weighed porcelain dish. The dish containing the sample was then placed inside the desiccator filled with the saturated salt solution. The desiccator was stored at room temperature and the sample was periodically weighed every 24 hours until a constant weight was reached, indicating that the equilibrium moisture content had been attained. A constant weight was determined when the deviation in weight from three consecutive weighinging for samples stored at relative humidity below 90% and above 90% did not exceed 2 mg/g and 10 mg/g,

respectively. Samples that had achieved a constant weight were then measured for their moisture content.

2.3.4 The determination of the isothermal sorption curve

The values of equilibrium moisture content derived from experiments were plotted against the relative humidity (RH) or water activity (aw) values in order to determine the isothermal sorption curve.

2.3.5 The determination of the isothermal sorption equation model

The determination of the isothermal sorption equation model was conducted to attain the highest degree of curve smoothness. Therefore, all three regions of the isothermal sorption curve are represented by the selected equation, which is applicable to food substances over the RH 0-95% range. In this study, five equation models were used:

$$\text{Chen Clayton: } a_w = \exp[-P1/\exp(P2*Me)] \quad (2)$$

$$\text{Oswin: } Me = P1[a_w/(1- a_w)]\ P2 \quad (3)$$

$$\text{Caurie: } \ln Me = \ln P1 - P2*a_w \quad (4)$$

$$\text{Henderson: } 1-a_w = \exp(-KMen) \quad (5)$$

$$\text{Hasley: } a_w = \exp[-P1/(Me)P2] \quad (6)$$

where; K and n = constant; P1 and P2 = constant, Me = Equilibrium moisture content; a_w = activity of water.

2.3.6 Model evaluation

Model evaluation was conducted to assess the accuracy of the selected isothermal sorption equation models in depicting the entire experimental isothermal sorption curve [20]. The evaluation involved calculating the Mean Relative Deviation (%MRD) for each model. The formula for %MRD is as follows:

$$MRD(\%) = \frac{100}{n}\sum_{i=1}^{n}\left|\frac{Mi - Mpi}{Mi}\right| \quad (7)$$

Where: Mi = Experimental moisture content data (%), Mpi = Calculated moisture content data (%), n = quantity of data, MRD< 5 = precise model; MRD<10 = almost precise model; MRD>10 = imprecise model.

2.3.7 The fitting of the slope value (b) of the isothermal sorption curve

The value of the slope (b) of the isothermal sorption curve was calculated inside the linear region. Labuza and Schmidl [21] determined the slope of the isothermal sorption curve by analyzing the linear region between the initial moisture content and the critical moisture content. The isothermal sorption curve was derived from the chosen isothermal sorption model. The Labuza equation was used to determine the slope value (b) and assess its influence on the shelf life of the product.

2.3.8 The determination of the weight of solids per packaging and the surface area of the packaging

Following the correction of the initial moisture content (Mo) with the initial weight of the product (Wo) in packaging, the result was represented as the weight of solids per packaging (Ws). The surface area of the utilized packaging (A) was determined through the multiplication of its length and width.

2.3.9 The determination of water vapor transmission

The characterization of water vapor was conducted based on gravimetry-based, evaluating the water vapor transmission rate using the wet cup method according to ASTM E 96-95 [22]. Each packaging was measured for its thickness. Petri dishes were measured for their surface area and filled with 30 mL of distilled water, then covered with aluminum foil. The aluminum foil was pierced to make holes covering 10% of the surface area of the distilled water. The packaging to be tested was used to cover the holes in the aluminum foil containing the distilled water, adhered with epoxy glue. The sealed petri dishes were allowed to stand for 10 minutes to ensure complete adhesion. The petri dishes were then weighed and positioned in a dehydrator at a temperature of 37 ± 0.5 °C. Samples were taken and weighed every hour for 5 hours. The values of water vapor transmission rate (WVTR) and water vapor permeability (WVP) were obtained using Equations 8 and 9, respectively.

$$\text{WVTR (g s}^{-1}\text{m}^{-2}) = \frac{The\ loss\ of\ moisture\ (g)}{Time\ (s) x\ wide\ area\ (m2)} \quad (8)$$

$$\text{WVP (g s}^{-1}\text{m}^{-1}\text{Pa}^{-1}) = \frac{WVP}{S\ x\ (R1-R2)} \text{ x d} \quad (9)$$

Where: S = saturated vapor pressure at a temperature of 37˚C (6266.134 Pa), d = film thickness (m), R1-R2 = RH in the cup (100%) = RH at 37˚ C (81%).

3 Results and discussion

3.1 Initial moisture (Mi) of surimi freeze-dried powder

The determination of initial characteristics of dried surimi involved proximate analysis and assessment of a_w and moisture content (MC) in the product. MC was a critical parameter for dried surimi, susceptible to fluctuations due to changes in environmental relative humidity (RH) during storage, as compared to other parameters. Alterations in moisture content directly impacted the water activity (a_w) and protein content of this hygroscopic surimi product. Hence, moisture content and water activity were identified as primary factors influencing the initial changes in dried surimi. The Mi content of surimi powder typically varied between 7-12%.

While there is no established commercial standard for surimi powder, the dried form of products can be evaluated by referring to the established standard for another seafood powder moisture content. The standard for fish protein concentrate is an alternative method that takes into account moisture content in addition to protein extraction. To prevent microbial changes in the product, the moisture content should be below 15%. The effectiveness of the moisture sorption isotherm in assessing the quality of freeze-dried tuna was also documented in a recent study by Rahmana [23]. This is because a_w is directly linked to the MC in the sorption isotherm, which determines the equilibrium moisture content. Furthermore, a_w is regarded as

a crucial characteristic in assessing the stability of foods relative to the overall water content. The assessment of food stability in terms of food texture, lipid oxidation, microbial growth, enzymatic, and non-enzymatic activities, and is frequently conducted using the concept of water activity [24].

The production of surimi powder with a moisture content of 15% or less is consistent with findings from a previous report, in which 4.6% moisture was present in freeze-dried tilapia surimi powder, according to a report by Ramirez et al. [25]. Surimi powder with an approximate 5% moisture content was also produced by using *Threadfin bream*, as reported by Huda et al [26]. Using various drying procedures, a comparable report obtained surimi powder with a concentration of 10.55%, which was produced from red sea bream surimi powder also reported by several researchers [27]. Eventually, although there are many factors that can influence the deterioration of surimi powder in this study, the initial change agreed upon from the previous report was moisture content. Thus, its change will be evaluated as the main parameter for further analysis using the Labuza model.

3.2 Critical moisture (Mc) of surimi freeze-dried powder

Utilizing the isothermal sorption curve method to ascertain the shelf life of dried products requires exact estimation of their critical moisture content (Mc). Dry surimi is an intermediate product, making hedonic parameters not directly applicable. Water activity and moisture content are considered as initial factors that can be observed and serve as determinants for the degradation of dry surimi. The critical moisture content of dry surimi in this study was determined based on a linear equation obtained from the curve correlation relating the logarithmic values of moisture content and water activity (a_w). A_w point of 0.80 was set as the rejection threshold for the surimi powder due to being the moisture content threshold for dry product quality.

The Mi content of dry surimi was 7.56%, and the critical moisture content in the study was 19.65% at a water activity of 0.807. The correlation linking storage duration and a_w values can be observed in Figure 1.

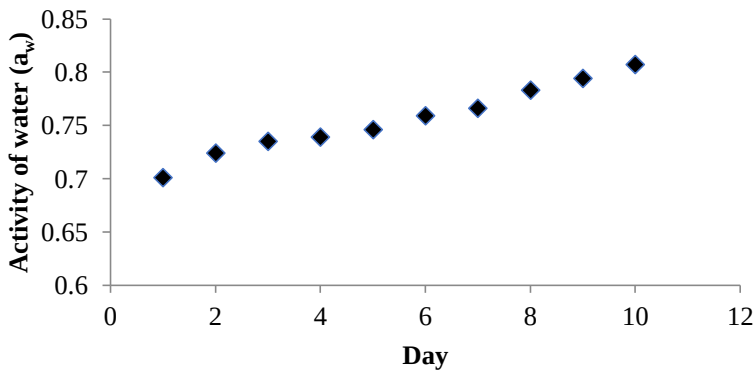

Fig. 1. The graph depicts the correlation between storage duration and a_w.

Figure 1 depicted the correlation between storage duration and a_w results. The water activity values for dry surimi increased with the duration of storage. The initial water activity value for dry surimi ranged around 0.7, categorized as a dry food product. The critical water activity value was determined at the upper limit of the water activity range for dry products, which was 0.80. The surimi powder was stored at room temperature without packaging, directly affecting its deterioration and water activity (aw) value. The critical moisture content

was reached at an aw of 0.8 within a 10-day experiment. Consequently, even if samples were stored beyond the 12-day period, the change in critical content was not significant to further evaluation, as the critical value had already been surpassed. The curve determining the critical moisture content based on water activity values can be observed in Figure 2.

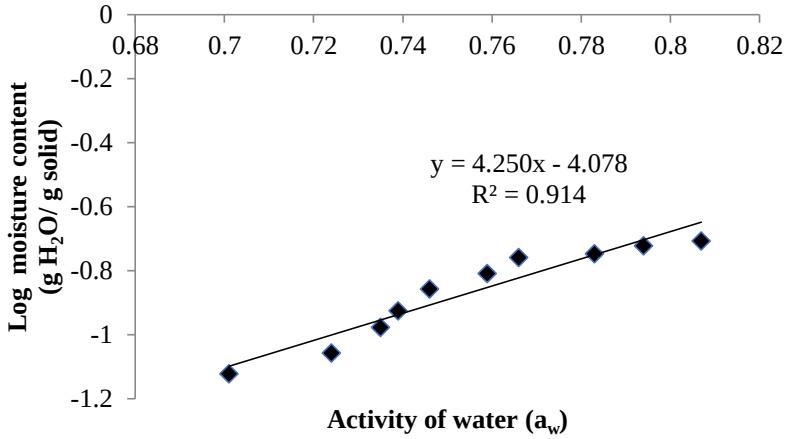

Fig. 2. The curve determines the critical moisture content of freeze-dried surimi based on water activity.

The Mc content was calculated by plotting the critical a_w value (0.8) into the linear regression equation derived from the curve correlation between the logarithmic moisture content and a_w values. The critical level was chosen as the minimum threshold for the water activity requirement, transitioning from dry food to semi-moist food. The obtained linear equation was $y = 4.250x - 4.078$ with an R^2 value of 0.914. The R^2 value indicated the precision in depicting the real conditions. The higher the value, the stronger the relationship between the two compared factors. According to the above calculation, the Mc content of the product could be determined, where $x = 0.80$, i.e., for dry surimi, it was 0.2098 g H_2O/g solid.

3.3 Equilibrium moisture content (Me) of freeze-dried surimi

The equilibrium moisture content (Me) of a surimi-based material refers to the water content at which the vapor pressure of its moisture is balanced with the vapor pressure of the surrounding environment. This occurs when the product no longer experiences an increase or decrease in weight. The determination of Me content involved storing freeze-dried surimi in a (modified) desiccator comprising a saturated salt solution with a specific relative humidity at room temperature. The product was stored under extreme and varied RH conditions, differing from the typical RH observed during storage. The use of 11 types of saturated salt solutions aimed to condition the RH within a 10% range.

The utilization of varying relative humidity (RH) values was intended to achieve the horizontal and most accurate isothermal sorption curvature (sigmoid) for deciding the shelf life of the product. During storage at different RH levels, molecular interactions occurred between water molecules in the product and the environment. This resulted in the transfer of water vapor either commencing the atmosphere into the surimi powder or contrarywise until a balance condition was attained. The transfer followed due to the difference between the RH of the environment and the a_w of the surimi powder, causing water vapor to move starting high relative humidity to low relative humidity. The Me value was achieved after the adsorption or desorption process.

Table 1. The Me and time required to attain those at different storage relative humidity (RH) levels of freeze-dried surimi.

Equilibrium RH (%)	Me (g H_2O / g solid)	Time (days)
6.90	0.099	22
22.60	0.129	20
32.40	0.152	18
43.00	0.154	16
57.50	0.197	14
64.00	0.217	13
69.00	0.251	11
75.50	0.203	9
84.00	0.176	9
93.00	0.496	12
97.00	0.664	12

Equilibrium conditions during storage were characterized by a constant increase or decrease in sample weight. The difference in sample weight had to be less than 2 mg/g for three consecutive weighing at relative humidity below 90% and less than 10 mg/g for three consecutive weighing at relative humidity above 90%. Changes in sample weight during storage indicated the hydration phenomenon. The rate of equilibrium attainment varied for each product and depended on the initial water activity conditions. At RH 60-70%, the product reached equilibrium faster, aligning with the initial water activity value of 0.701 for dry surimi. The relative humidity in the range of 90% then reached equilibrium on day 12, followed by salts with lower RH values until day 22 at RH 6.9%.

3.4 Curve and model of the isothermal sorption of freeze-dried surimi

The isothermal sorption curve was a graph illustrating the correlation between water activity (aw) or equilibrium relative humidity (ERH) and the moisture content per gram of a surimi-based material. The water sorption isotherm graph for freeze-dried surimi (Figure 3) is presented below.

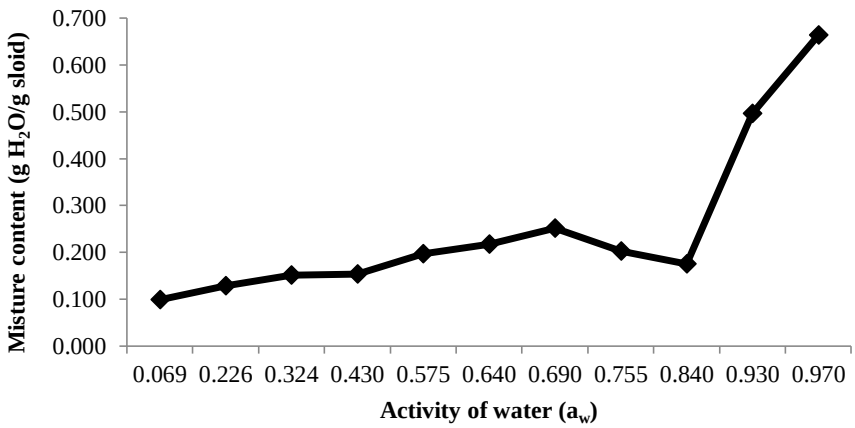

Fig. 3. The graph depicts the correlation between a_w and Me content (ISA) of freeze-dried surimi.

The Me content acquired from the experiments was marked against the a_w or ERH values to create a curve known as the isothermal sorption curve. The resulting curve typically exhibits an S-shaped pattern (sigmoid), which is distinctive and characteristic for each food material. The isothermal sorption curve is valuable for depicting the water content of the material concerning storage conditions. The unique shape of the ISA curve was attributed to differences in physical structure, chemical composition, and water-binding conditions within food materials. This curve illustrates the water adsorption and desorption phenomena in surimi materials, making it widely used in determining shelf life, storage, packaging, and drying processes. The isothermal sorption curve also describes the hydration process concerning chemical interactions of water molecules on the surface, the release of molecular structures to accelerate transfer, and volume changes due to open molecules [28].

The hysteresis phenomenon explains that different water activity values are obtained for measurements of foods with the same moisture content, depending on how the moisture content is achieved, through adsorption or desorption processes. Generally, the model of the isothermal sorption curvature is specific to each material. Isothermal sorption can describe the characteristics of food materials and provide information about the relative conditions of microbial attack during storage [29]. The isothermal sorption curve can also depict the water content of the material concerning storage conditions.

These nonlinear models were then converted into linear equations (y = a + bx) to facilitate calculations. The steady points were calculated by the least squares method, which selects the best regression line among all possible straight lines that can be drawn on a scatter plot. The linear equations derived from the isothermal sorption curve models can be seen in Table 2.

Table 2. The models for the isothermal sorption curve of freeze-dried surimi.

Model	Linear Equation (y = a + bx)	MRD
Hasley	$\log Me=(\log(\ln(1/a_w))+1.893)/-2.209$	2.310
Chen Clayton	$Me=(\ln(\ln(1/a_w))-0.733)/-6.83$	9.784
Henderson	$\log Me=((\log(\ln(1/(1-a_w)))-1.059)/1.704$	5.634
Caurie	$\ln Me = -2.509 + 1.629\ a_w$	4.098
Oswin	$\ln Me = 1.707 + 0.297 \ln (a_w/1-a_w))$	2.516

The above models were then utilized to determine the Me content of freeze-dried surimi. The precision of the isothermal sorption curvature in depicting the sorption phenomena was assessed based on the convergence of the experimental isothermal sorption curve with the curves obtained from the equations. The accuracy of these equation models was further assessed by calculating the mean relative determination (MRD) values [30]. The accuracy test of the isothermal sorption equations aimed to determine the precision of the selected models, thus obtaining an isothermal sorption curve via the MRD estimation.

The equation model that most accurately depicted the isothermal sorption curve for freeze-dried surimi was the Hasley model. The Hasley equation was selected as the fit model with the curve that utmost closely matched the experimental isothermal sorption curve compared to other equation models. The Hasley equation model exhibited the lowest MRD value among the equation models, which was 2.31. This result indicated that the Hasley equation model could precisely depict the entire isothermal sorption curve of freeze-dried surimi (MRD < 5). Similar findings were reported by Limonu [31], who also identified the Hasley model with the least MRD number in defining the shelf life of corn powder. The Caurie and Oswin equation models also accurately depicted the entire isothermal sorption curve (MRD < 5), while the Chen Clayton and Henderson equation models still had MRD values > 5. The equation for the Hasley model for the isothermal sorption curve of freeze-

dried surimi was log Me = (log(ln(1/a_w))+1.893)/-2.209. The isothermal sorption curve displayed on the selected model for freeze-dried surimi can be seen in Figure 4.

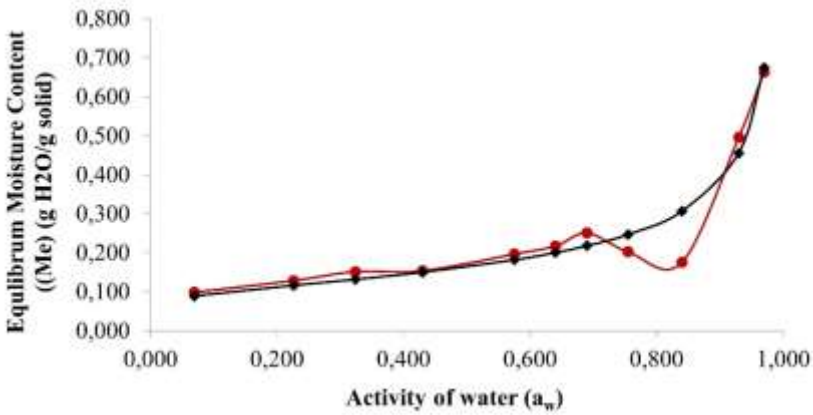

Fig. 4. The isothermal sorption curve based on the Hasley model (●) and experimental results for surimi powder (●)

Based on the obtained water activity (a_w) values, it was concluded that surimi products were on the verge of safety limits for processing into certain food products, reaching their critical moisture content. These values fell within the critical range for the growth of harmful microorganisms such as molds, yeasts, and bacteria. Some mold types, such as Mucor, Neurospora, and Rhizopus, tended to grow rapidly in high-moisture food materials but were not harmful if the water activity of the food was below 0.90. Xerophilic molds generally thrived at water activity levels below 0.85. All xerophilic mold types were known to produce mycotoxins at water activity levels around 0.75 [32]. Based on the observations from the determination of the isothermal curve, surimi powder stored in highly humid conditions (>90% RH) experienced mold growth after a storage period of two months. This represented the initial storage condition that led to a deterioration in physical quality.

3.5 The value of the slope (b) for the isothermal sorption curve of frozen surimi powder

The slope (b) of the sorption isotherm curve was calculated from the linear region. The linear zone was assessed by comparing the Mi content area with the Mc content, as shown in Figure 4. The link between a_w and Me value can be described by a linear equation of the form y = a + bx. The variable b in this equation denotes the gradient of the sorption isotherm curve.

Slope 1 represented the slope obtained from the experimental results, while Slope 2 was derived from the calculations. The value of Slope 1 for dry surimi was 1.416, calculated based on the alteration between the Mi content and the Mc content, as well as the difference between the initial water activity and the critical water activity. Slope 2 was obtained from the linear equation formed on the Hasley model isothermal sorption curve, passing through the initial water activity. The slope 2 of the Hasley model isothermal sorption curve was 0.478.

3.6 The acceleration method for determining the critical water content via the isothermal sorption curve approach

The prediction of shelf life employing the isothermal sorption curve approach was conducted by storing dried surimi at commonly used storage relative humidity (RH), which was at 70% and 90% RH. These RH ranges were chosen as they were commonly employed in the storage of food products in tropical regions. The research laboratory was maintained at room temperature conditions during storage with an 85% RH, based on measurements from the Darmaga Climatology Station in Bogor, Indonesia. The relatively high environmental humidity was attributed to the research period conducted during the rainy season (February-March). The relative humidity (RH) of the environment significantly influenced shelf life. Higher RH contained more water vapor, leading to increased water vapor absorption into food products compared to lower RH. The greater absorption of water vapor accelerated deterioration, resulting in a shorter shelf life for the product. The calculated parameters for determining the shelf life of dried surimi can be found in Table 3.

The minimum shelf life for dried surimi with HDPE packaging at 90% RH was one month, while the maximum shelf life was obtained for dried surimi with retort pouch packaging, reaching 22.6 months at 70% storage RH. This aligned with the research conducted by [33], who found that dried tuna packaged with aluminum had a longer shelf life (91 months) compared to dried tuna packaged with HDPE, which had a shelf life of only 40 months. As the storage relative humidity increased, the pressure difference also increased. The pressure difference was influenced by environmental RH and product RH. Product RH could be calculated through the measured product water activity. Therefore, the pressure difference was also influenced by the product water activity, which may change during storage. The use of retort pouch packaging indicated the longest shelf life for dried surimi due to its low permeability compared to OPP and HDPE. Considerations for packaging selection also involved distribution time and economic factors for the final dried surimi product. The same trend was also reported in every commodity, revealing different and unique sorption curve models, making it more significant to evaluate and assess during the shelf-life study assessment [34-35].

Table 3. Determination parameters of the shelf life of freeze-dried surimi under different packaging and relative humidity (RH)

Parameter	OPP		HDPE		Retort Pouch	
	RH 70%	RH 90%	RH 70%	RH 90%	RH 70%	RH 90%
Mi (g H_2O)/g solid)	0.0697	0.0697	0.0697	0.0697	0,0697	0,0697
Mc (g H_2O)/g solid)	0.2099	0.2099	0.2099	0.2099	0,2099	0,2099
Hasley Model = log Me=(log(ln(1/aw)) + 1.893) /-2.209						
The slope of the sorption isotherm curve (b)	1.416	1.416	1.416	1.416	1.416	1.416
Equilibrium Moisture content (Me) (g H_2O/g solid)	0.222	0.385	0.222	0.385	0.222	0.385
Permeability of the packaging (k/x) (g/m^2.day.mmHg)	0.074	0.074	0.100	0.100	0.020	0.020
Surface area of the packaging (A) (m^2)	4.2	4.2	4.2	4.2	4.2	4.2
Weight of solids per packaging (Ws) (g)	500	500	500	500	500	500
Vapor pressure at temperature 30 °C (Po) (mmHg)*	31.824	31.824	31.824	31.824	31.824	31.824
Ln (Me-Mi)/(Me-Mc)	2.556	0.588	2.556	0.588	2.556	0.588
A/Ws	0.0084	0.0084	0.0084	0.0084	0.0084	0.0084
Po/b	22.478	22.478	22.478	22.478	22.478	22.478
Shelf-life (day)	183	42	135	31	677	156
Shelf-life (month)	6.1	1.4	4.5	1.0	22.6	5.2

*Referred to the American Meteorological Society standard [36].

4 Conclusion

Based on the experiment calculating the shelf life of frozen dried surimi employing the critical moisture content model, a smooth ISA curve was obtained with 11 saturated salts. The Hasley equation was selected as the preferred model in determining the shelf life of dried surimi. According to the Labuza equation, the estimated shelf life of surimi at 70% and 90% RH was 6.1 and 1.4 months for surimi with OPP plastic packaging, 4.5 months and 1 month for surimi using HDPE plastic, and 22.6 months and 5.2 months for surimi with retort pouch packaging. Since the initial change in moisture content serves as the primary indicator of deterioration in surimi powder, it is important to conduct further evaluations encompassing physical, chemical, nutritional, sensory, and microbiological aspects to accurately determine its shelf life. Additionally, introducing RH control aids and other dependent variables could potentially enhance and prolong the quality of dried surimi before its commercialization.

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