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Water sorption isotherm of Aceh Rice (*Oryza sativa*): Study on chemical properties and characteristics

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Abstract The chemical properties and isothermic characteristics of water absorption in Aceh rice was investigated. The results showed that Aceh Mutan M4 local rice had 3 bound water fractions, namely 7.1% db, 12.3% db and 169.5% db. Type of water bound to the area of Primary Bonded Water (PBW), Secondary Bonded Water (SBW), and Tertiery Bonded Water (TBW) of Aceh local rice were Mp = 7.1% db, Ms = 12.3 % db, and Mt = 169.5% db. Storage of rice for 15 days in the ATP area of product quality did not damage or change, storage in ATS became older in color, while storage in ATT areas decreased product quality which was indicated by the presence of fungus. The implication of this research suggested that local Aceh rice should be stored at a water content of <7.1%, because the first critical points of Aceh local rice was Mp 7.1% db and equilibrium ap = 0.53.

Keywords: Agrotechnology, Chemical, Isotherm, Mutation, Rice

Introduction

Rice is the main food commodity of Indonesian society which is used as a carbohydrate requirement for the community. The increase in the population of Indonesia causes an increase in the demand for national rice. The national population in 2015-2020 increased by 2.36%. Data from the Central Statistics Agency shows that the national rice consumption per week per capita in 2021 was 2,551 kg or an increase of 0.06 kg from 2020 (Munawar and Sabaruddin, 2021).

Local rice is naturally resistant to pests and diseases, tolerant of abiotic stress, and has good quality rice so that it is liked by the people in the locations where it grows and develops. Local cultivars are seen as very valuable assets and need to be managed properly. Upland rice cultivation on dry land is largely determined by varieties that are adaptive to these conditions (Savari *et al.*, 2020). Until now, the available upland rice varieties are very limited, so it is

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necessary to assemble superior upland rice varieties that are adaptive to dry land. The assembly of new varieties can be done through plant breeding activities. Agricultural commodities are naturally hygroscopic, that is, they can absorb water from the surrounding air and on the contrary can release some of the water contained therein into the surrounding air. both before and after processing (Jia *et al.*, 2016; Xu *et al.*, 2016).

These hydratation properties are illustrated by the water sorption isotherm curve, which is a curve that describes the relationship between the moisture content of the material and the relative humidity of the space where the material is stored or water activity (aw) at a certain temperature (Afandi *et al.*, 2016; Qiang *et al*., 2014). Awanthi *et al.* (2019) attempted to apply this water sorption isotherm to describe water in maintaining the stability of food and agricultural products during storage. This sorption isotherm curve is used as the basis for determining the physico-chemical properties of an agricultural commodity and its processed materials. that is, a curve that describes the relationship between the moisture content of the material with the relative humidity of the space where the material is stored or water activity (aw) at a certain temperature (Das *et al.*, 2020; Zhang *et al.*, 2019).

Previous studies attempted to apply this water sorption isotherm to describe water in maintaining the stability of food and agricultural products during storage. This sorption isotherm curve is used as the basis for determining the physico-chemical properties of an agricultural commodity and its processed materials. that is, a curve that describes the relationship between the moisture content of the material with the relative humidity of the space where the material is stored or water activity (aw) at a certain temperature. Water sorption isotherm describe water in maintaining the stability of food and agricultural products during storage. This sorption isotherm curve is used as the basis for determining the physico-chemical properties of an agricultural commodity and its processed materials (Chuang *et al.*, 2014; Siriphollakul *et al.*, 2017; Zhang *et al.*, 2011).

Water in food and agricultural products can be classified into 2 types, namely bound water and free water. The properties of free water in foodstuffs are the same as those of ordinary water in general with a value of aw = 1, while bonded water is water that is closely tied to other foodstuff components and has aw below 1. Water sorption isotherm curves in food are generally sigmoid in shape and can be related to different water activities on solids. Xu *et al.*, (2018) reported the existence of three fractions of bonded water in dry matter, namely primary bonded water (ATP), secondary bonded water (ATS) and tertiary bonded water (ATT), while Munawar and Sabaruddin (2021) distinguished it from monolayer water (type I), multilayer water (type II) and free moving water (type III). The time interval from production to refusal of food is said to be shelf life. Some of the factors that influence shelf life are product characteristics, environment and packaging properties. Determination of product shelf life can be done using the ESS method (Extended Storage Studies), ASS (Accelerated Storage Studies) and the ISA analysis method.

The values of aw and Me are variables that can be used for predictive analysis of food damage and determining the drying time required for product stability (Qingyun *et al.*, 2007). Water activity (aw) related to a minimum water content of the agricultural products that caused mircoba to live. Furthermore, the moisture equilibrium (Me) related to the optimal moisture contents for storage in preserving agricultural products quality. Foodstuffs greatly determine the conditions of absorption or loss of water from food, so a mathematical model is developed that can be used to predict the shelf life of a product. The quality of rice is influenced by several main factors such as genetics, pre-harvest activities and the environment, harvesting and post-harvest treatments (Johnson *et al.*, 2019a; Li *et al.*, 2008).

Rice quality can be based on market-based quality, rice quality and health quality. Market-based quality consists of physical quality and milled quality. Physical quality includes seed length and shape, moisture content, seed appearance, whiteness, and liming grain. Local rice varieties are rice varieties that have long adapted to certain areas. The use of local rice is generally used as food in the form of rice. Rice varieties used were Sigupai varieties with 19 genotypes. The purpose of this study was to determine the chemical properties and isothermic characteristics of local Aceh rice water absorptionwords.

Materials and methods

The material used in this study was the Sigupai variety rice using 9 lines. The rice used for this research was obtained from the Faculty of Agriculture, Syiah Kuala University. The chemicals used are K2CO3, NaBr, NaNO2, KI, SrCl2, NaNO3, KBr, desiccators, aluminum foil, and lime. 2.1. Analysis of chemical properties: water content (Hayati *et al.*, 2021; Munawar and Sabaruddin, 2021), fat content, protein content (Jiang *et al.*, 2020), ash content, carbohydrate content (Bao *et al.*, 2007; Johnson *et al.*, 2019b). Preparation of rice at 2%, Preparation of saturated salt solution, Each saturated salt solution was prepared as much as ± 100 ml for each desiccator. The sample (0.6 g) was put into an aluminum foil plate and equilibrated in a desiccator. The balance of moisture and rice content is carried out in a desiccator containing a saturated salt solution and tightly closed. The desiccator was stored in an incubator at a temperature of 28 C, and every day the sample was weighed until the moisture content was equal. 2.3. Measurement of balance water content, Data were analyzed using the BET equation (Brunauer, Emmet, Teller) to produce primary bonded water (ATP). ATP can be determined based on the BET water absorption isotherm mathematical model, with model aw/ (1-aw) M = 1 / Mpc + (c-1) / Mpcaw. By means of BET, the first critical water content (Mp). The logarithmic model equation to produce Secondary Bound Water (ATS) is to obtain water activity (aw) critical and critical relative humidity (RH). Secondary bound water (ATS) or the second water fraction is a multilayer water layer whose analysis can use a semilogarithmic mathematical model, with a model, - Log (1-aw) = p + q (M). With this model a second critical water content (Ms) and the second critical water activity (as). Tertiary Bonded Water (ATT) is carried out by determining the limit value of tertiary bound water with free water (Mt) carried out through 2 approaches, first the extrapolation method of the order 2 polynomial model and the second the manual extrapolation method, using the concept of free water with a value = 1.

Results

Chemical properties of rice resulted to the F test of variance analysis that showed that the genotype treatment of Aceh local rice of mutant M4 as seen in Table 1.

Table 1. Average chemical properties of Aceh local rice

Treatment	Water content	Fat level	Protein content	Ash content	Carbohydrate levels
Genotype					
G1	13,98de	1,24b	5.02bc	1.17a	78.59b
G2	14.17bcd	1.42ab	4,94bc	1.12a	78.35b
G3	14.03cde	1,21b	4.88c	1.17a	78.70b
G4	14.43ab	1,22b	4,80cd	1.12a	78.43b
G5	14.02cde	1.32ab	5,21ab	1.18a	78.28bc
G6	14.58a	1.32ab	5,36a	1.11a	77.63d
G7	14.13cde	1,51ab	5,36a	1.17a	77.82cd
G8	14.27bc	1.66a	4,55d	1.12a	78.41b
G9	13,88e	1.55ab	3,79e	1.17a	79.60a
BNJ (5%)	0.277	0.410	0.305	0.077	0.481
Land					
Gogo	14,17tn	1.77a	4,16b	1.16a	78.75a
rice fields	14,17tn	1.00b	5.60a	1,14b	78.10b
BNJ (5%)	0.080	0.119	0.089	0.022	0.139

Information: numbers followed by different letters in the same column show a real difference based on the results of the honest real difference test at the 5% real level

The relationship between water content and aw is described in terms of the sorption isotherm curve as presented in Figure 1. Water sorption isotherm is an important characteristic that can affect aspects of drying and storage. Water sorption isotherm shows the relationship between the water content of the material and the RH of the equilibrium of the space in which the material is stored or the activity of water at a certain temperature. The shape of the water sorption isotherm in general would determine the stability of the storage. Water sorption isotherm curves were used to determine the shelf life using the ASS (accelerated storage studies) method, namely the storage of food products in higher environmental conditions than normal storage conditions. The advantage of this method required a short testing time and had high accuracy and accuracy. In food ingredients, water sorption isotherm can describe the water content of the material as the relative humidity of the storage space.

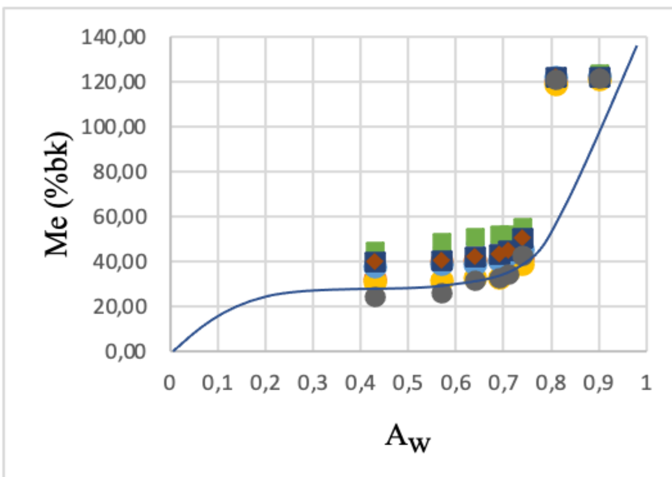

Figure 1. Aceh Mutan M4 local rice water sorption isotherm curve at room temperature 28 ^{o}C

The purpose of the water sorption isotherm is to determine the water content critical to the longevity of the M4 mutant Aceh rice. From this critical water, we get water, primary bound, secondary bound water, tertiary bound water. The results of the upland water sorption isothermic curve and the rice siren sorption isothermic curve, the curve obtained showed a sigmoid-shaped upward curve although not perfect, where the desorption process showed the behavior, namely the higher the equilibrium relative humidity, the higher the equilibrium water content (Me) would also be conversely afftecd the lower the equilibrium relative humidity, the lower the equilibrium water content would also be faced. Reduced water content in the desorption process indicated water diffusion from rice seeds to the environment.

Primary bonded water

To determine the critical water content, the ISA data analysis was used with a modified BET mathematical model, which only applies to the aw 0 range - 0.60 (Soazo et al., 2011), with equation (1):

$$\frac{a_w}{(1-a_w)M} = \frac{1}{M_pC} + \frac{c-1}{M_pC}a_w \text{ .... (1)}$$

Where M is water content (%), c is constant, Mp is the capacity or limit of primary bound water (%). Mp is the first critical water content. Equation (1) can be viewed as linear regression with the independent variable aw. The results of the regression analysis and the Mp value are shown in Table 2.

Table 2. Regression equations and primary bound water boundary for Aceh local rice

Strains	Regression Equations	R2	Point a	Value b	Value c	Mp (%) bk
1	Y = 0.1974x-0.0867	0.8636	a = 1 / Mpc	b = (c-1) / Mpc	b / a = (c-1)	9.0
2	Y = 0.192x-0.0784	0.8956	a = 1 / Mpc	b = (c-1) / Mpc	b / a = (c-1)	8.8
3	Y = 0.1842x-0.0791	0.8804	a = 1 / Mpc	b = (c-1) / Mpc	b / a = (c-1)	9.5
4	Y = 0.2496x-0.109	0.8672	a = 1 / Mpc	b = (c-1) / Mpc	b / a = (c-1)	7,1
5	Y = 0.2065x-0.0886	0.877	a = 1 / Mpc	b = (c-1) / Mpc	b / a = (c-1)	8.5
6	Y = 0.1637x-0.0682	0.903	a = 1 / Mpc	b = (c-1) / Mpc	b / a = (c-1)	10.0
7	Y = 0.1925x-0.0817	0.8692	a = 1 / Mpc	b = (c-1) / Mpc	b / a = (c-1)	9.0
8	Y = 0.2507x-0.1058	0.9188	a = 1 / Mpc	b = (c-1) / Mpc	b / a = (c-1)	10.0
9	Y = 0.1917x-0.0698	0.9198	a = 1 / Mpc	b = (c-1) / Mpc	b / a = (c-1)	8.1

Information: Y = regression equation, a = regression constant value a, b = regression constant value b, c = obtained from constants a and b, Mp = primary bound water limit, bk = dry weight

Secondary bonded water

Secondary bound water analysis (ATS) used water content data above ATP. To determine ATS, a logarithmic analysis model was used. To determine the secondary bound water content (Ms), a semilogarithmic analysis model is used with the general equation (2):

- log (1-aw) = p + q (M) .... (2)

Where M is water content (%) in water activity aw, p and q are linear regression constants.

The equation data plot produces a straight line that breaks into two straight lines. The first straight line represents the area of the secondary bound water fraction, namely at the moisture content of the range 24.8-35.4% and the second straight line represents the tertiary bound water fraction, namely the area of water content in the range of 35.4-55.5%, with model equations and results. The regression analysis of the equation is shown in Table 3. The intersection point of the two broken lines is the point of transition from secondary to tertiary bonded water and is seen as the upper limit or capacity of secondary bond water. It would produce the intersection point of the two lines which is the boundary of the second and third water fraction areas and the value is called the second critical water content (Ms) as shown in Figure 2. From this equation, the aw boundary between the primary and secondary bound water fraction areas is the first critical water activity (ap) and the aw boundary between the secondary and tertiary bound water fraction areas, namely the second critical aw as follows:

$p1 + q1\ Ms = p2 + q2\ Ms$ .... (3)

Table 3. Equations of Secondary Tied Water and first and second critical water activities of Aceh local rice

Strains	Equal	Me% bk	ap	US	RH
G1	p1 + q1Ms = p2 + q2Ms	12.4	-	-	
	-log (1-ap) = p1 + q1Mp	-	1.69	-	169
	log (1-as) = p2 + q2Ms	-	-	0.60	6
G2	p1 + q1Ms = p2 + q2Ms	6.4	-	-	
	-log (1-ap) = p1 + q1Mp	-	1.62	-	162
	log (1-as) = p2 + q2Ms	-	-	0.37	37
G3	p1 + q1Ms = p2 + q2Ms	4,2	-	-	
	-log (1-ap) = p1 + q1Mp	-	1.67	-	167
	log (1-as) = p2 + q2Ms	-	-	0.30	30
G4	p1 + q1Ms = p2 + q2Ms	9,1	-	-	
	-log (1-ap) = p1 + q1Mp	-	0.53	-	53
	log (1-as) = p2 + q2Ms	-	-	0.38	38
G5	p1 + q1Ms = p2 + q2Ms	4	-	-	
	-log (1-ap) = p1 + q1Mp	-	0.67	-	67
	log (1-as) = p2 + q2Ms	-	-	0.42	42
G6	p1 + q1Ms = p2 + q2Ms	14.6	-	-	
	-log (1-ap) = p1 + q1Mp	-	0.65	-	65
	log (1-as) = p2 + q2Ms	-	-	0.60	6
G7	p1 + q1Ms = p2 + q2Ms	0.48	-	-	
	-log (1-ap) = p1 + q1Mp	-	0.65	-	65
	log (1-as) = p2 + q2Ms	-	-	0.15	15
G8	p1 + q1Ms = p2 + q2Ms	12.3	-	-	
	-log (1-ap) = p1 + q1Mp	-	2,4	-	240
	log (1-as) = p2 + q2Ms	-	-	0.34	34
G9	p1 + q1Ms = p2 + q2Ms	2,4	-	-	
	-log (1-ap) = p1 + q1Mp	-	1.5	-	150
	log (1-as) = p2 + q2Ms	-	-	0.62	62

Note: Ms = second and third water fraction boundary (second critical water content), ap = first critical water activity, as = second critical water activity

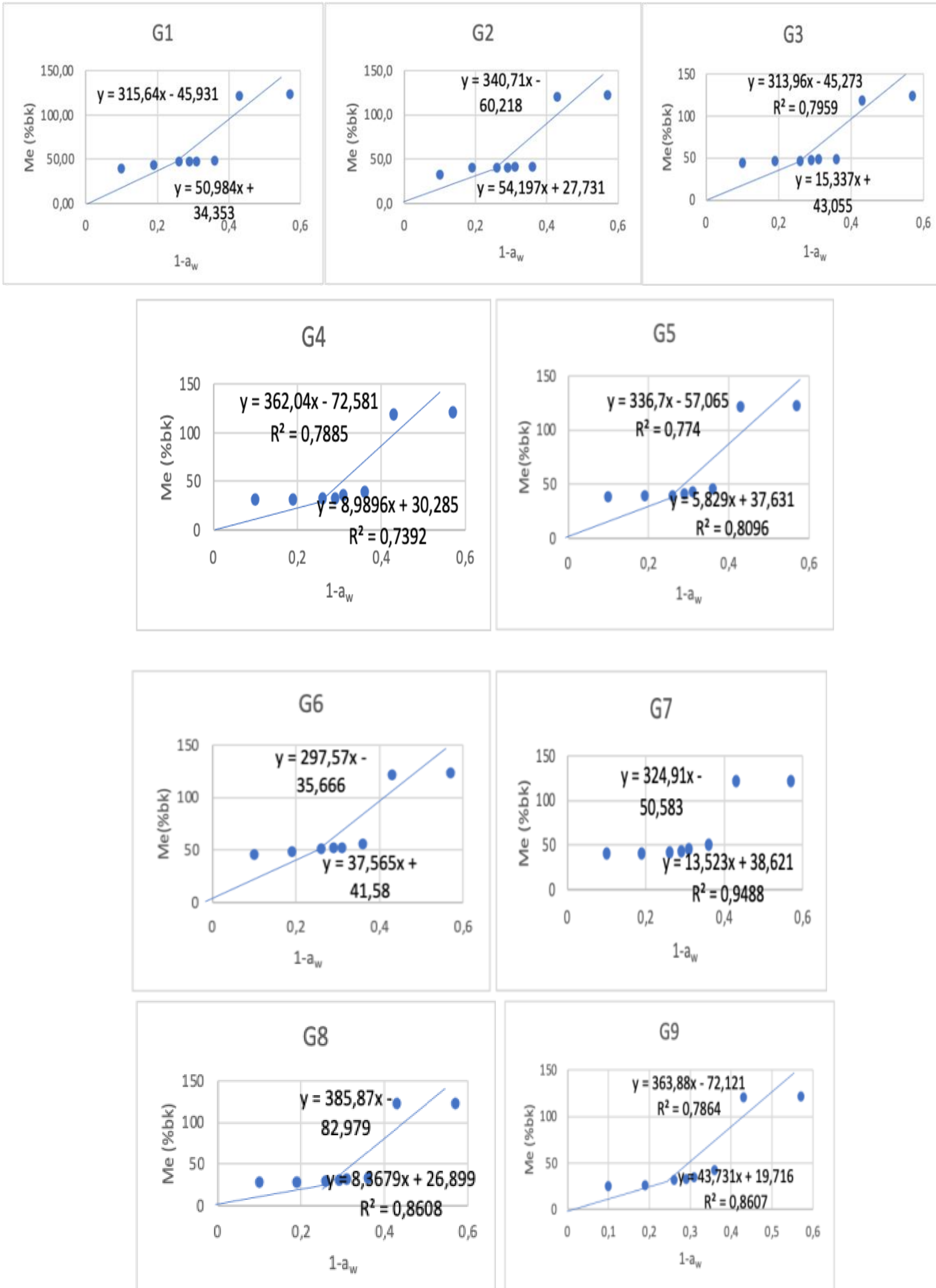

Figure 2. Secondary bound water curves in local rice Aceh Mutan M4

Water activity is very important and is associated with the stability or deterioration of dry products. If a chemical reaction occurs in the second water fraction area, the dry product damage by microbial growth occurs in the third water fraction area. Water activity can indicate the lowest limit for the growth of microbe resistant to halophilic salts (aw 0.60), most molds (aw 0.80), yeast (aw 0.86) and pathogenic bacteria (aw 0.91).

Tertiary bonded water

Tertiary bound water is used when it damaged to the product which is indicated by the presence of microbial growth in a product. To determine the tertiary bound water limit, ISA data from the tertiary bound water area are used, namely the water content of 12.3% bk and above. Based on the concept that free water has aw value = 1, the extrapolation method either regression or manual can be used. With the regression analysis method used the order 2 polynomial model.

By entering the value x = aw = 1 in the regression equation, the upper limit value of the tertiary bound water fraction (Mt = Y (x = 1)) is obtained which becomes the equation:

$$Y = ax\ 2 + bx + c \text{ .... } (4)$$

Where is Y water content and x water activity. From the regression equation, it is obtained the boundary water boundary value for local Aceh rice tertiary is shown in Table 4.

Table 4. Regression equation, tertiary bound water from Aceh local rice

Strains	Regression Equations	R2	Mt (%) bk
G1	Y = -353.19x3 + 1417x2-1206.7x + 327.04	0.8228	184.2
G2	Y = -52,898x3 + 865,36x2-872.62x + 254.53	0.816	194.4
G3	Y = -189.12x3 + 1129x2-1059.6x + 308.76	0.8359	189
G4	Y = -699.54x3 + 2270.8x2-1849.2x + 465.29	0.8322	187.4
G5	Y = -818.86x3 + 2445.1x2-1928x + 483.09	0.8271	181.3
G6	Y = -1131.8x3 + 3031.9x2-2304.3x + 574.66	0.8483	169.5
G7	Y = -908.9x3 + 2585.4x2-1995.4x + 495.13	0.8354	176.2
G8	Y = -914.83x3 + 2802.5x2-2251.5x + 556.77	0.8165	192.9
G9	Y = -1214x3 + 3252.7x2-2429.7x + 567.62	0.8493	176.6

Note: Mt = tertiary bound water limit

Determination of the tertiary bound water limit can also be done manually, namely seeing the lowest value obtained from Table 5 and producing an Mt of around 169.5%.

Discussion

The results of the analysis on local rice mutant M4 Aceh had a water content with genotype G6 significantly higher with a value of 14.58% compared to genotype G9 with a value of 13.88%. According to SNI No. 6128 of 2015 concerning rice, the maximum water content standard for rice is 14 percent. Rice moisture content that was more than 14 percent causes faster damage during storage. During storage, the moisture content of the rice is maintained, so that it is not too high to prevent the growth of fungi and change the rice structure to become brittle or break. At high water content, the texture of rice is relatively soft and breaks easily. This also in agreement with Bingol *et al.* (2012) mentioning that in high water vapour content, might make them more susceptible to fissuring and tending to break rapidly. Rice planted in upland and paddy fields had no significant yield, so it does not affect the yield.

The results of the analysis of fat content in the mutant rice M4 genotype G8 were significantly higher with a value of 1.66% compared to other genotypes, but these results were not significantly different from G2, G5, G6, G7, and G9, while the lowest was in G3 value 1.21%. Other findings reported A different procedure was used for samples with higher water content (moisture content: 0.28e0.72 g/g, web basis) and these samples contained freezable water (Wan *et al.,* 2018). The fat content of rice ranges from 0.58-1.23 percent in milled dry rice and 2.4-3.9% in skin-cracked rice and the fat content is the second and third composition of spread in rice. The fat content planted by rice on upland land gave significantly higher yields than rice planted in the fields. Similar findings also noted that different landuse, climate and altitude may had impact on paddy-rice yields (Kaur *et al.,* 2021; Toğrul and Arslan, 2006; Witczak and Gałkowska, 2021).

Aceh local rice from mutant M4 genotypes G6 and G7 was significantly higher with a value of 5.36% compared to G5 with a value of 5.21%. The lowest genotype with a value of 3.79% for genotype G9. Rice with high protein content produces creamy rice color and smells bad. The tendency to increase protein content was due to the long drying treatment, it can be concluded that the longer drying time, the increased protein content (Camaño Echavarria *et al.,* 2021; Chen *et al.,* 2019; Mallek-Ayadi *et al.,* 2020). The curve pattern of water sorption normally as sigmoid. Similar findings also noted by Bingol *et al.* (2012) and Sahu and Patel (2020) that moisture sorption isotherms of different forms of M202 and M206 exhibited the sigmoid (Type II) shape. Furthermore, Protein content in rice is influenced by the genotype, plant cultivation system, and the analytical method used. The protein content planted in paddy fields gave significantly higher yields than rice grown on upland land.

Analysis of the variety in ash content in the local aceh mutant rice from M4 showed that there was not significantly affected. Ash content analysis is used to determine whether or not a processing process was good to know the type of material used, to determine or distinguish original or synthetic materials, as a parameter of the value of foodstuffs. Ash content planted with rice in upland received a very high yields compared to rice planted in paddy fields. Similar findings also noted and compared in literatures that ash and carbogydrates content are generated different yields due to environmental effect of paddy-rice cultivation (Sahu and Patel, 2020; Torres and Seijo, 2016; Wan *et al.*, 2018).

Carbohydrates are nutrients that can be found in the largest quantities in rice. Carbohydrates in cereals, including rice, are mostly in the form of starch. The determination of carbohydrate content in the proximate analysis is carried out by difference. The total amount of water, ash, fat, protein and carbohydrates in rice was 100%. The results of the carbohydrate analysis examined the genotype average ranged of 77.63% to 79.60%. The highest average value of carbohydrates was 79.60%, while the lowest was 77.63%. According to literature, the carbohydrate content of rice was in the range of 78%.

Based on obtained results, it may conclude that Aceh Mutan M4 local rice has 3 bound water fractions, namely 7.1% db, 12.3% db and 169.5% db. The types of water bound to the ATP, ATS, and ATT areas of the M4 mutant Acehnese local rice are Mp = 7.1% db, Ms = 12.3% db, and Mt = 169.5% db, respectively. Storage of rice for 15 days in the ATP area of product quality did not experience damage or change, storage in ATS only became older in color, while storage in ATT areas occurred product damage which was indicated by the presence of mold. It is recommended that local Aceh rice should be stored at a water content of <7.1%, due to the first critical points of M4 mutant Acehnese local rice are Mp 7.1% db and equilibrium ap = 0.53. The second critical points were Ms = 12.3% db and equilibrium as = 0.34 where at the first and second critical points there had not been damaged to local rice mutant M4 Aceh.

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