# Storage of Wheat Malt: Sorption Properties and Water Activity Interrelations with Malt and Wort Characteristics

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#### **ABSTRACT**

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The interrelation of water activity ( $a_w$ ) and wheat malt and wort characteristics during storage was investigated. The sorption isotherm of wheat malt was sigmoid. For storage, equilibrated malts corresponding to  $a_w$  of 0.112, 0.328, 0.52, and 0.756 were kept at 30°C and analyzed for changes in malt and wort characteristics at bimonthly intervals for eight months and at the end of 12 months. On storage, malts corresponding to  $a_w$  of 0.112 and 0.328 showed some variation in diastatic power and  $\alpha$ -amylase activity. Malts kept at 0.52  $a_w$  showed minimal changes in diastatic power

and  $\alpha$ -amylase activity and at the same time gave higher extracts. Except for the yield of extract, which was satisfactory, deterioration in malt characteristics occurred when malts were stored at 0.756 a<sub>w</sub>. Analysis of data by response-surface methodology, applying second- and third-order polynomials, and by a linear correlation coefficient matrix showed that decreasing pH of worts caused by increasing a<sub>w</sub> of the malts affected wort characteristics considerably more than the duration of storage.

There are a few studies regarding the storage quality of malts. Storey et al (1977) investigated the effects of storage atmosphere and selective relative humidities (15 and 65%) on barley malt and wort characteristics. Deteriorative changes in malt quality were reported at the higher relative humidity of 65% under airflow atmosphere. Literature on the storage quality of barley malt is somewhat perceptive (Schuster 1962). Barley is customarily used for malting and brewing. Wheat malt has a potential for exploitation, due to its ready availability for use as food malt (Pomeranz et al 1975, Sethi and Bains 1978, Singh and Sosulski 1985). Storage quality of wheat malt has not been studied so far. The present investigation was aimed at delineating the interrelationship of water activity (a<sub>w</sub>) and duration of storage on the wheat malt and wort characteristics.

#### MATERIALS AND METHODS

## Preparation of Malt

A sample of *Triticum aestivum* cv. WL 1562 was obtained from the research farm of the Punjab Agricultural University, Ludhiana, India. Weighed lots (5 kg) of the grain were steeped at 15°C to a moisture content of 44% (Singh and Sosulski 1985). The drained, steeped grain was germinated for five days at 15°C and 95% relative humidity. The green malt was dried in a through-flow air drier at 55°C for 20 hr followed by kilning at 85°C for 4 hr. The malt contained 4% moisture and was kept in an air-tight container after removing the roots.

#### **Sorption Characteristics**

The sorption isotherm of the wheat malt was determined gravimetrically at 30°C (Lang and Steinberg 1981), and monolayer moisture determined according to Langmuir (1918). Weighed aliquots (about 2 g) were kept in 3-cm-diameter petri dishes in desiccators containing saturated salt solutions (Hall 1957) providing water activities (aw, % equilibrium relative humidity/100; Labuza 1968) ranging from 0.112 to 0.863. Each desiccator holding one set of duplicate samples was placed in a temperature-controlled cabinet. The samples were weighed at intervals of 24 hr until equilibrium moisture was attained, when two consecutive weighings showed no change in weight.

#### Storage

Separate lots of malt (750 g) were kept in desiccators containing saturated salt solutions of LiCl, K<sub>2</sub>CO<sub>3</sub>, MgNO<sub>3</sub>, and NaCl at 30° C

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giving  $a_w$  values of 0.112, 0.328, 0.52, and 0.756. Equilibrated malts corresponded to 2.9, 6.5, 9.0, and 14.0% moisture, respectively. Equilibrated malt lots were homogeneously mixed and aliquots withdrawn for analysis. A concurrent control in an air-tight container was kept in a refrigerator at  $4 \pm 2^{\circ}C$ .

#### Malt and Wort Analyses

Diastatic power (DP) of malts in degrees Lintner (°L) was determined by the method of AOAC (1980).  $\alpha$ -Amylase activity was determined by the ICC method (Perten 1966) using  $\beta$ -limit dextrin substrate. The results were expressed in Sandstedt, Kneen, and Blish units (SKB units/g).

The malt samples were mashed according to the AOAC method (1980). An iodine staining test was used to determine the conversion time (min). The yield of extract (%) was determined from the specific gravity (20/20° C) equivalent of the filtered wort from the Plato tables (52.009, AOAC 1980). Wort soluble nitrogen (N) and color were determined by the AOAC procedures (1980). Color was expressed as absorbance at 430 nm. The reducing sugars in worts (% maltose) were determined by using 3,5-dinitrosalicylic acid reagent (Bernfeld 1955). Titratable acidity (% lactic acid) and pH were estimated by the ASBC methods (1958), and free  $\alpha$ -amino N (mg/L) determined using ninhydrin reagent (Lie 1973). Modification index was calculated by expressing the soluble N as percent of total N in malt. The samples were analyzed in triplicate and mean values reported.

## Statistical and Response-Surface Analyses

The results were examined statistically using the factorial design of the experiment  $(4 [a_w] + 1 [control] \times 5 [time])$  and the Hewlett Packard computer, HP 1000 system. The analysis of variance was performed in a one-way classification. The least significant difference (LSD) test was used to judge the significance of difference between the means (Steel and Torrie 1960). Linear correlation coefficients (r) were calculated between the variables and relationships developed using the 2647 I terminal of Hewlett Packard graphics plotter. Response-surface methodology was applied for analysis of the data (John and Quenouille 1977). Changes in malt and wort characteristics with changes in  $a_w$  and storage time were examined by second- and third-order polynomials.

#### **RESULTS AND DISCUSSION**

#### Sorption Characteristics

The sorption isotherm of wheat malt is shown in Figure 1. The monomolecular layer of moisture was formed with about  $0.03\,\mathrm{g}$  of water per gram of dry solid (Langmuir 1918) when exposed to the atmosphere of  $0.112\,\mathrm{a_w}$ . Additional layers of water were adsorbed by the malt kernels placed in the atmospheres ranging from  $\mathrm{a_w}~0.22$ 

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to 0.52. The moisture content of malt increased as  $a_w$  exceeded 0.52 and revealed adsorption of water in multimolecular layers. At  $a_w$  of 0.52 the malt kernels retained a crisp and friable texture desirable for grinding. This information formed the basis for storing wheat malt at different  $a_w$  atmospheres.

#### Effect of Storage on Malt Quality

The mean values for various characteristics of freshly prepared wheat malt and worts are shown in Table I. The diastatic power of control malt stored for two months (Fig. 2) was similar to that of the fresh malt. Some decrease in the values occurred as storage was extended to four months. However, between four and 12 months, there was negligible difference in diastatic power of control malt. The malts kept at 0.112 and 0.328 a<sub>w</sub> showed variable values for diastatic power, which leveled off at that of the control malt after storage for 12 months. Lots exposed to 0.52 a<sub>w</sub> showed minimal changes in diastatic power. Increased a<sub>w</sub> of the storage atmosphere to 0.756 substantially decreased the diastatic power, except for those of the six-month stored malts. Fluctuation in the values for diastatic power of stored malts was possibly the result of some changes in biochemical components of the malt.

The  $\alpha$ -amylase activity was similar in malts stored for two to eight months at 0.112, 0.328, and 0.52  $a_w$  (Fig. 3). The values for four- and six-month malts were significantly (P < 0.05) lower than those of the control malts. Extended storage of 0.112- and 0.328  $a_w$ -malts to 12 months increased the  $\alpha$ -amylase activity considerably, whereas that of the 0.52  $a_w$ -malt decreased marginally. Large decreases in  $\alpha$ -amylase activity occurred in malts stored at 0.756  $a_w$ . According to Storey et al (1977), the brewers' malt stored at 65% relative humidity (0.65  $a_w$ ) for 12

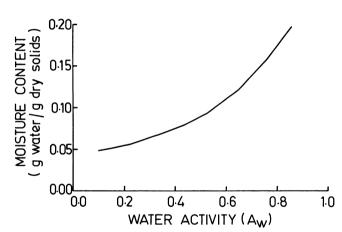


Fig. 1. Sorption isotherm of wheat malt at 30° C.

TABLE I
Initial Composition of Wheat Malt and Wort

Composition	$\mathbf{Mean} \pm \mathbf{SD^a}$		
Malt <sup>b</sup>			
Diastatic power (° L)	$137 \pm 1$		
$\alpha$ -Amylase activity (SKB units/g)	$90.4 \pm 5.4$		
Conversion time (min)	4		
Hot water extract (%)	$76.5 \pm 0.1$		
Soluble nitrogen (%)	0.90		
Modification index <sup>c</sup> (%)	40.9		
Wort <sup>d</sup>			
Color (absorbance at 430 nm)	0.23		
рH	5.9		
Titratable acidity (% lactic acid)	0.10		
Reducing sugars (% maltose)	$5.1 \pm 0.3$		
Free α-amino nitrogen (mg/L)	$155 \pm 10$		

<sup>&</sup>lt;sup>a</sup> Mean of three replications.

d As is.

months showed a decline in  $\alpha$ -amylase activity.

Higher extract yields (3-6%) were obtained from malts stored for two months (Fig. 4) than those of the fresh malt (Table I). Although differences in hot water extract of malts stored for two to eight months were small, the malts of 0.112, 0.328, and 0.756  $a_w$  showed fluctuations in extract yields, whereas the changes in those of 0.52  $a_w$  wheat malts were negligible. Prolonged storage of malts for 12 months at  $a_w$  of 0.52 and 0.756 increased the extracts

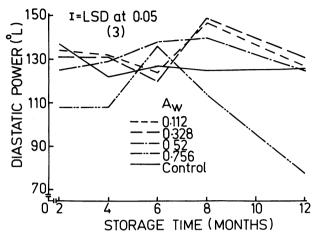


Fig. 2. Effect of storage and water activity  $(a_w)$  on diastatic power  $(^{\circ}L)$  of wheat malts.

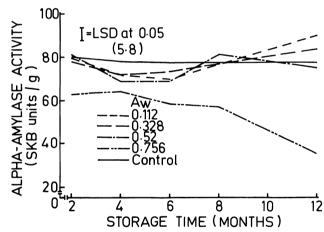


Fig. 3. Effect of storage and water activity  $(a_w)$  on  $\alpha$ -amylase activity (SKB units/g) of wheat malts.

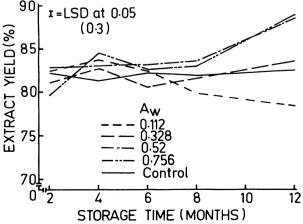


Fig. 4. Effect of storage and water activity  $(a_w)$  on yield of hot water extract (%) of wheat malts.

<sup>&</sup>lt;sup>b</sup>Dry basis.

 $<sup>^{</sup>c}$ (Soluble N/malt N)  $\times$  100.

perceptibly, despite their lower  $\alpha$ -amylase activities, compared with malts kept at 0.112 and 0.328 a<sub>w</sub>. Storey et al (1977) observed a decrease in extract yield for brewers' malt stored at 65% relative humidity for 12 months.

The soluble N contents of the malts stored at 0.52 and 0.756 aw were higher than those of the control and the malts stored at 0.112 and 0.328 aw (Fig. 5). The amount of soluble N was affected less by storage from two to 12 months. However, sharp increases in soluble N in worts were observed for malts corresponding to aw of 0.52 and 0.756 after storage for six months. The degree of modification was thus increased compared with that of fresh malt (Tables I and II). A linear correlation between the values for modification index versus soluble N of malts was statistically

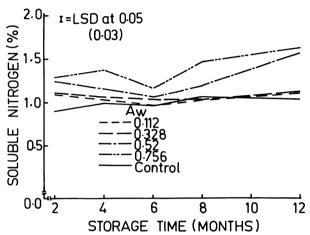


Fig. 5. Effect of storage and water activity (a<sub>w</sub>) on soluble nitrogen (%) in wheat malts.

TABLE II Effect of Water Activity (a<sub>w</sub>) and Storage on Modification Index of Wheat Malt

	Modification Index <sup>a</sup> (months of storage)						
a <sub>w</sub>	2	2 4 6 8					
Control <sup>b</sup>	40.9	45.0	44.6	47.1	49.0		
0.112	51.9	46.8	46.8	48.6	51.2		
0.328	49.3	50.6	47.9	45.8	51.0		
0.520	59.2	53.4	49.3	55.4	64.9		
0.756	60.0	59.7	50.4	64.9	72.0		
LSD (0.05)	7.8	7.8	7.8	7.8	7.8		

<sup>&</sup>lt;sup>a</sup> Modification index = (soluble N/wort N)  $\times$  100.

highly significant (Table III). Differences in the values for modification index of control malts and those stored at 0.112 and 0.328 aw were small, except for malt stored for two months. On increasing the aw to 0.52 and 0.756 for storing malts, a notable increase in the degree of modification was observed.

#### Effect of Storage on Wort Quality

Conversion time of stored malts during mashing varied from 3 to 10 min. However, the malts stored at 0.756 a<sub>w</sub> for 12 months took a considerably longer time for conversion, which was ascribed to decreased  $\alpha$ -amylase activity (Fig. 3). Worts from malts kept at 0.112 and 0.328 aw showed no more discernible change in pH compared with the control (Table IV). A larger decrease in wort pH was observed in malts kept at 0.52 and 0.756 a<sub>w</sub>, especially when those malts were stored for 12 months. The color (absorbance at 430 nm) of worts from the control and 0.112 a<sub>w</sub>-malts was comparable and was of light hue (Table IV). An inverse and significant coefficient of correlation between color and pH of worts was obtained (Table III). The color values also correlated well with diastatic power,  $\alpha$ -amylase activity, soluble N, and modification index of the stored malts. Evidently, decreased pH of worts catalyzed Maillard-type reactions (Hodge and Osman 1976). As storage aw was increased from 0.112 to 0.756, color increased for worts and was more pronounced as storage was extended to 12 months. During storage browning of malt kernels was also observed from darkened appearance of adhering embryo.

Differences in the reducing sugar values for worts of malts stored in the range of  $a_w$  from 0.112 to 0.756 were negligible, except for those of the malts stored two months (Table IV). Worts from stored malts had more reducing sugars than those of the control malts. Titratable acidity values for worts of malts stored at 0.112 and 0.328  $a_w$  conditions were similar to those of the control (Table IV). Worts from malts kept at 0.52 and 0.756  $a_w$  showed higher values for titratable acidity, which increased further with storage from two to eight months and then decreased when tested after storage for 12 months, probably due to metabolic transformations. The free  $\alpha$ -amino N values for worts of malts corresponding to 0.112, 0.328, and 0.52  $a_w$  showed fluctuations as storage extended from two to 12 months (Fig. 6). Worts of 0.756  $a_w$ -malts showed higher free  $\alpha$ -amino N values.

# Interrelationships Between aw, Storage, and Malt and Wort Characteristics

The regression coefficients obtained by response-surface methodology using second- and third-order polynomials showed significant relationships between storage variables and malt and wort characteristics (Table V). Response-surface methodology is most often based on a first- or second-order polynomial. The results of third-order polynomials explained more elaborately the curved response surface than the quadratic terms. Evidently, the values for regression coefficients obtained from second-order polynomials were considerably lower than those of the third order.

TABLE III

Linear Correlation Coefficients for Malt and Wort Characteristics<sup>a</sup>

Malt/Wort A	Malt			Wort					
	AAA	EY	SN	MI	pH	CO	TA	TRS	FANb
Malt									
Diastatic power	0.789**°	-0.447**	-0.654**	-0.662**	0.582**	-0.792**	-0.201	-0.107	-0.513**
α-Amylase activity (AAA)		-0.526**	-0.626**	-0.563**	0.670**	-0.899**	-0.615**	-0.357**	-0.534**
Extract yield (EY)			0.668**	0.640**	-0.425**	0.576**	0.222	0.191	0.245*
Soluble N (SN)				0.982**	-0.862**	0.777**	0.470**	0.351**	0.707**
Modification index (MI)					-0.467**	0.739**	0.415**	0.338**	0.697**
Wort									
pН						-0.836**	-0.676**	-0.282*	-0.792**
Color (CO)							0.563**	0.319**	0.670**
Titratable acidity (TA)								0.556**	0.554**
Total reducing sugars (TRS)									0.390**

n = 75.

<sup>&</sup>lt;sup>b</sup>Malt kept at 4°C.

 $<sup>^{</sup>b}$ FAN = Free  $\alpha$ -amino nitrogen.

<sup>&</sup>lt;sup>c</sup>\* and \*\* = Significant at the 5 and 1% levels, respectively.

The results of cubic correlations summarized in Table V revealed considerably more influence of aw on malt and wort characteristics as compared to storage time. The effect of aw conditions on pH of wort, degree of modification, and soluble N, color, and titratable acidity was pronounced. Diastatic power, hot water extract, and reducing sugars in worts were comparatively less affected, whereas  $\alpha$ -amylase activity and free  $\alpha$ -amino N were intermediately affected. Decrease in wort pH with increased aw of stored malts was probably due to progressive increase in the acid phosphatase activity in the aleurone cells of malt kernels with accompanying release of organic/inorganic phosphates (Ashford and Jacobson 1974). Labuza (1971) reported increased enzyme activity with increase in a<sub>w</sub> of food systems. It is possible that the phosphate ions react with divalent ions, partly during storage and partly during mashing, to liberate H<sup>+</sup> ions and result in decreasing wort pH below those of malts at lower a<sub>w</sub>. The dissociated PO<sub>4</sub><sup>3-</sup> moiety is also likely to increase titratable acidity in worts. This was

TABLE IV Effect of Water Activity (a<sub>w</sub>) and Storage on Wheat Malt Wort Characteristics

			Stora	ge (mo		
Characteristic	$\mathbf{a_w}$	2	4	6	8	12
pH	Control	5.90	5.85	5.83	5.72	5.70
•	0.112	5.68	5.87	6.01	5.74	5.70
	0.328	5.68	5.88	5.98	5.72	5.70
	0.520	5.55	5.76	5.62	5.48	5.40
	0.756	5.23	5.37	5.34	4.90	4.71
LSD (0.05)	•••	0.01	0.01	0.01	0.01	0.01
Color	Control	0.23	0.23	0.21	0.27	0.23
(absorbance at 430 nm)	0.112	0.22	0.25	0.23	0.23	0.24
,	0.328	0.28	0.26	0.25	0.24	0.26
	0.520	0.29	0.28	0.27	0.30	0.39
	0.756	0.51	0.70	0.65	1.00	1.70
LSD (0.05)	•••	0.01	0.01	0.01	0.01	0.01
Total reducing sugars	Control	5.1	5.2	5.2	6.6	6.7
(% maltose)	0.112	5.3	6.9	7.4	7.0	7.1
,	0.328	6.0	6.9	7.4	7.0	7.2
	0.520	6.5	6.9	7.5	7.1	7.2
	0.756	6.6	7.0	7.7	7.1	7.3
LSD (0.05)	•••	0.3	0.3	0.3	0.3	0.3
Titratable acidity	Control	0.10	0.12	0.12	0.13	0.11
(% lactic acid)	0.112	0.08	0.11	0.14	0.14	0.10
,	0.328	0.10	0.12	0.14	0.15	0.11
	0.520	0.11	0.13	0.17	0.18	0.14
	0.756	0.15	0.18	0.23	0.27	0.16
LSD (0.05)	•••	0.01	0.01	0.01	0.01	0.01

<sup>&</sup>lt;sup>a</sup> Malt kept at 4°C.

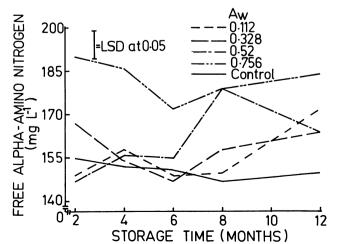


Fig. 6. Effect of storage and water activity  $(a_w)$  on free  $\alpha$ -amino nitrogen (mg/L) in wheat malt worts.

TABLE V
Regression Coefficients as Calculated by Response-Surface Methodology
for Water Activity (a<sub>w</sub>), Storage, and Wheat Malt
and Wort Characteristics<sup>a</sup>

	Polynomial Order						
Malt/Wort	а	w	Storage				
Characteristic	Second	Third	Second	Third			
Malt							
Diastatic power	0.371	0.561** <sup>b</sup>	0.325*	0.417*			
α-Amylase activity	0.399*	0.676**	0.129	0.204			
Hot water extract	0.433*	0.472**	0.361	0.437*			
Soluble N	0.599**	0.797**	0.366*	0.378			
Modification index	0.581*	0.782**	0.378*	0.414			
Wort							
рH	0.522**	0.810**	0.289	0.397*			
Color	0.426*	0.708**	0.298	0.298			
Titratable acidity	0.503*	0.704**	0.560*	0.577			
Total reducing sugars	0.514*	0.575**	0.572*	0.573			
Free α-amino N	0.428*	0.678**	0.218	0.224			

 $<sup>^{</sup>a}n = 75$ 

supported by significant inverse correlation between pH and titratable acidity values for the worts (Table III). The effect of aw on soluble N might be an artifact of wort pH. According to Briggs et al (1981), the pH optima for the activity of proteases during mashing is between pH 4.6 and 5.0. The malts stored at 0.52 and 0.756 a<sub>w</sub> had more soluble N than those corresponding to 0.112 and 0.328 aw (Fig. 5). It is likely that the protease activity in malts stored at the higher aw increased due to decreased wort pH (Table IV), thereby increasing the soluble N level in worts. This is further confirmed by an inverse and significant correlation between soluble N and wort pH (Table III). Increased carboxypeptidase activity at decreased pH of wort (Briggs et al 1981) was shown by the values for higher free  $\alpha$ -amino N in worts, especially in 0.756  $a_w$ -malts (Fig. 6). Significant inverse correlation was obtained between free  $\alpha$ -amino N and pH of wort (Table III). Because the effect of aw on malt during storage was less on diastatic power, hot water extract and reducing sugars in worts indicated that the endosperm part of the malt was affected to a lesser extent by aw of malt compared with the aleurone layers.

#### CONCLUSION

Wheat malts stored at  $0.52~a_w~(30^\circ\,C)$  minimally changed diastatic power and  $\alpha$ -amylase activity. Malts containing equilibrated moisture contents of up to 9% as maximum corresponding to an  $a_w$  of 0.52 at  $30^\circ\,C$  were optimal for about six months storage and retained desirable malt and wort quality. The response-surface methodology analysis of the data revealed greater response to  $a_w$  conditions in relation to malt and wort characteristics than the duration of storage. Thus, a latitude of up to 9% moisture content in wheat malt is commercially advantageous from the standpoint of storage stability.

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b\*, \*\* Significant at the 5 and 1% levels, respectively.

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