

TEXTILE TECHNOLOGY

Method for the Prediction of the Rate of +b Color Change in Upland Cotton (*Gossypium hirsutum* L.) as a Function of Storage Temperatures

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ABSTRACT

Previous studies have indicated that HVI +b values of baled Upland cotton increase due to prolonged storage with uncontrolled temperature conditions. This change in +b is accompanied by decreases in reducing sugar content and pH. The present study attempts to elucidate the dependence of this trend on storage temperature and time, and to develop a model capable of predicting +b changes at any given temperature and time. A single bale of cotton was subjected to varying temperature and time conditions, and resultant values of reducing sugar content, pH, and +b were determined. Exponential regressions gave rate constants that were temperature dependent and obeyed the Arrhenius expression. A model was constructed that allows prediction of +b as a function of time and temperature. The model will be used as a basis for the construction of a more extensive model that can be used to predict color change in any Upland cotton based on initial +b, reducing sugar content, and pH values.

The market value of raw cotton is determined by a number of critical factors, including fiber length uniformity, fiber strength, leaf grade, and color grade. Color grade is classed by the Agricultural Marketing Service using HVI (Uster Technologies, Knoxville, TN), and is based on measured Rd (diffuse reflectance) and +b (yellowness) values. Although few published scientific studies have been performed demonstrating that cotton yellowness affects yarn processing, yarn quality, or finished fabric dyeability, it is widely accepted that color is an indicator for potential difficulties, including the appearance of barré (Bailey, 2002). A correlation between the increase in cotton yellowness due to warehouse ageing and a decrease in yarn strength

has been reported (Gamble, 2007a). Furthermore, decreases in reducing sugar levels were significantly correlated with both decreasing yarn strength and increasing fiber yellowness. Even though yellowness in raw cotton is used to set market value, the causes of it have been investigated only in a limited number of studies. Previous studies (Marsh et al., 1958) have indicated that field-weathered cottons exhibiting relatively low copper reduction values, which are indicative of low reducing sugar content, display a lower propensity to brown as a result of heating at 160 °C. The authors attributed this lowered sugar content to the growth of microbes on the cotton surface. It was further established that decreasing reducing sugar content due to microbial growth was correlated with increasing pH levels.

A more recent study (Gamble, 2007b) has established that increases in cotton yellowness as a result of accelerated ageing at elevated temperatures not only accompanied by decreases in reducing sugars, but also by decreases in pH. This is a strong indication that the color changes produced in cotton under typical storage conditions (at or below 7% moisture content and temperatures not controlled) are due to a nonmicrobial induced mechanism. A chemical yellowing mechanism in cotton was proposed by Marsh, who suggested that the reaction in cotton is similar to browning reactions known to occur in other agricultural and food products. Such browning reactions may be due to enzymatic browning (Martinez and Whitaker, 1995), as in the case of sliced fruits and vegetables, or to nonenzymatic processes. The most common of these nonenzymatic browning reactions is caramelization (Kroh, 1994), which is a sugar dehydration reaction occurring at temperatures >100 °C, and the Maillard reaction (Nursten, 1981), which results from the chemical condensation between a reducing sugar and an amino acid. The Maillard reaction may occur at much lower temperatures than caramelization, and in many instances leads to deterioration in quality in a wide range of products, including processed fruit juices (Burdurlu and Karadeniz, 2003) and dairy formulations (Dattatreya et al., 2007). It is also the primary mechanism responsible

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for imparting the brown color to cooked meats (Bordas et al., 2004), baked bread (Mundt and Wedzicha, 2007), and fried potatoes (Quayson and Ayernor, 2007). Results of the previously cited study (Gamble, 2007b) have demonstrated that heating greige cotton yarn results in a decrease in reducing sugars and an increase in absorbance due to browning products. These changes were accompanied by a decrease in pH and an increase in fluorescence intensity. All of these factors have been extensively cited (Nursten, 2005) as indicators of the Maillard reaction. Based on these results, it is probable that the yellowing mechanism producing increases in +b in cotton is the Maillard reaction. Various techniques, including addition of sulfur dioxide, buffering at low pH, and refrigeration have demonstrated effectiveness at slowing the reaction (Nursten, 2005). Whether these methods are effective or economically viable treatments of baled cotton have not been extensively explored. It may be more effective to manage the storage conditions of cotton to minimize the rate at which the cotton undergoes an increase in +b. Prior to the last several years, storage conditions would not have been expected to affect the quality characteristics to the extent that they do today. The reason for this is the shift in consumption from domestic markets to overseas. This entails, in many cases, extended storage in warehouses at elevated temperatures or residence in cargo containers that likewise may expose the cotton to elevated temperatures for extended periods. The goal of the present study is to develop a methodology by which the rate of color change in stored cotton may be predicted as a function of storage temperature. In this study, samples from one bale of cotton were subjected to different temperature conditions for varying lengths of time. Resultant chemical and color data was used to develop a model to predict color changes.

MATERIALS AND METHODS

Accelerated ageing with added moisture. Fifteen separate 10 g samples from one bale of Upland cotton grown in New Mexico in the 2007 crop year were cleaned on a Shirley Analyzer (Shirley Institute, Manchester, England) and conditioned at 25 °C and 65% RH for 24 h prior in preparation for accelerated ageing using varying amounts of excess water. An initial moisture content of 6.7% (wet basis) of the cotton was measured according to standard test methods (ASTM, 2001). Three samples were subsequent-

ly placed in glass jars with tight fitting lids without additional moisture. Three identical sets were also prepared with added moistures of 1, 2, and 3% (wet basis), respectively, resulting in samples of 7.7, 8.7, and 9.7% moisture (wet basis). One additional set without added moisture was prepared and placed in jars without being sealed. These prepared samples were then placed in a laboratory oven at 60 °C. The samples were individually removed from the oven at different time intervals ranging from 24 to 96 h.

Accelerated ageing without added moisture. Similar 10 g samples were prepared and placed in glass jars without added moisture and sealed. Samples were treated at 50, 60, or 70 °C for times ranging from 2.5 to 418 h.

Color measurement. Following removal of the cotton samples from the oven, the jars were allowed to cool to room temperature and equilibrated at 25 °C and 65% RH for 24 h; after which they were subjected to HVI color measurements for Rd and +b according to standard test methods (ASTM 1999).

Sugar and pH determinations. Subsequent to HVI measurements, two separate 1 g samples were taken from each of the oven-treated samples and extracted using 20 ml deionized water per gram of cotton. Each sample was agitated with a glass rod to promote wetting of the cotton surface. The wetted sample was allowed to sit for 15 min before being wrung out and the extract was analyzed for pH using an Orion Model 310 pH meter (Orion Research, Inc., Beverly, MA). The same extract was then analyzed for glucose and fructose content using High Performance Anion Exchange Chromatography (Dionex Model ICS3000, Sunnyvale, CA). Each sugar was quantified based upon comparison of peak areas with the peak areas of a calibration solution containing 100 ppm of each sugar.

Statistics. Linear regressions were performed using SigmaPlot 8.0 (SPSS Science, Chicago IL). T-tests were performed using SigmaStat 3.0 (SPSS Science, Chicago IL).

RESULTS AND DISCUSSION

The rate of Rd and +b color change at 60 °C was determined as a function of added moisture content. The results indicate that +b changes over time are not dependent upon the amount of vapor phase moisture available (Table 1). However, the unsealed samples showed a much lower rate of change in +b. Assuming that the change in +b is due to the Maillard reaction,

the results are consistent with studies by Eichner and Karel (1972) that indicated that the reaction, though generally more efficient at lower water activities, slowed down under extremely dry conditions due to the impediment to mobility of the reactants. It was assumed for the purposes of this study that cotton stored in warehouses displays a water activity similar to the sealed samples in Table 1. Furthermore, because added moisture has no apparent effect on the rate of +b change, all subsequent results are reported for cotton samples that were sealed during heating with no added moisture.

Chemical and colorimetric data are presented in Table 2 for samples heated for varying lengths of time at three different temperatures. Both fructose and glucose contents decreased as a function of heating time at all of the observed temperatures;

in addition, pH decreased and +b increased. These results are consistent with previous results (Nursten, 2005) that demonstrated that the reaction between reducing sugars and proteins or free amino acids led to a decrease in pH as well as the production of colored compounds. At 50 °C, Rd does not exhibit a significant change, whereas at 60 and 70 °C, Rd decreases. These results suggest that under the conditions of this study, the change in Rd may be due to an alteration of the surface waxes, because the onset of wax melting is in the 60 to 70 °C range. This conclusion is in keeping with previously reported results (Gamble, 2007a) indicating that in the range of normally encountered cotton bale storage conditions (< 50 °C), Rd was not significantly affected. Therefore, this study was primarily concerned with changes to +b.

Table 1. Rd and +b as a function of moisture and heating time at 60 °C

Heating Time (h)	Added Moisture (% wet basis)									
	0		1		2		3		unsealed (dry)	
	+b	Rd	+b	Rd	+b	Rd	+b	Rd	+b	Rd
0	7.5(0.5)	80.4(0.8)	7.5(0.5)	80.4(0.8)	7.5(0.5)	80.4(0.8)	7.5(0.5)	80.4(0.8)	7.5(0.5)	80.4(0.8)
24	9.9(0.9)	79.1(1.4)	10.1(0.7)	78.6(1.5)	10.0(0.9)	78.3(1.3)	10.0(0.9)	77.8(1.3)	7.8(0.7)	79.9(1.2)
48	11.0(0.9)	78.3(2.0)	10.8(0.8)	77.5(1.6)	10.9(0.7)	76.7(0.9)	10.9(0.9)	76.0(1.6)	8.2(0.6)	80.1(1.4)
96	11.9(0.8)	75.8(0.9)	11.9(0.7)	75.3(1.1)	11.6(0.7)	74.4(0.7)	11.8(0.8)	74.2(0.7)	8.5(0.7)	78.8(0.9)

Table 2. Chemical and color variables as a function of heating time at 50, 60, and 70 °C^z

T (°C)	time (h)	glucose (ppm)	fructose (ppm)	pH	+b	Rd
50	0	986 (84)	700 (68)	7.25 (0.04)	7.5 (0.5)	80.4 (0.8)
	24	878 (88)	666 (94)	6.98 (0.03)	8.9 (0.6)	79.5 (0.8)
	72	792 (16)	610 (32)	6.88 (0.05)	9.9 (0.9)	79.8 (0.8)
	139	652 (2)	560 (28)	6.84 (0.02)	10.8 (1.0)	79.4 (1.2)
60	0	986 (84)	700 (68)	7.25 (0.04)	7.5 (0.5)	80.4 (0.8)
	24	776 (62)	628 (90)	6.89 (0.03)	10.0 (0.8)	78.5 (1.3)
	48	692 (2)	600 (44)	6.87 (0.02)	10.9 (0.8)	77.1 (1.7)
	96	540 (44)	572 (2)	6.76 (0.05)	11.9 (0.8)	74.9 (1.0)
70	0	986 (84)	700 (68)	7.25 (0.04)	7.5 (0.5)	80.4 (0.8)
	2.5	872 (34)	680 (38)	7.01 (0.03)	8.4 (0.7)	79.3 (0.8)
	24	664 (106)	580 (8)	6.76 (0.05)	11.2 (0.6)	75.7 (0.8)
	92	384 (2)	292 (2)	6.35 (.04)	13.3 (0.2)	71.0 (0.3)
	256	224 (32)	198 (84)	6.30 (0.02)	15.3 (1.3)	71.2 (1.8)
	418	110 (1)	108 (4)	6.27 (0.02)	16.7 (1.3)	69.2 (1.2)

^z Values in parentheses denote standard deviation

The relationship between $+b$ and total reducing sugar content of the heat-treated samples are shown in Fig. 1. When subjected to linear regression, the resulting fit strongly suggests an inverse linear relationship, with a slope of -162 , a y -axis intercept of 2965 , an x -axis intercept of $+b = 18.18$, and a coefficient of determination of $R^2 = 0.99$. This linear relationship suggests that the rate of change of $+b$ may be approximated as being of the same reaction order with respect to glucose and fructose. Determination of reaction orders in homogeneous systems with high water activities are relatively straightforward, being based on chemical kinetic theory, but become complex when the system in question displays multiple reaction pathways, i.e., both glucose and fructose reacting with any number of free amino acids or proteins. Adding to the complexity is the fact that these reactions may deviate from first order, making solution to the n^{th} order reactions ($n \neq 1$) much more difficult to attain than with first order reactions. It has been shown (Eichner and Karel, 1972) that in solid phase systems with low water activity, as in the present work, classical chemical kinetics does not provide a straightforward solution. With this in mind, an initial assumption of first order behavior with respect to the sugars was tested. The results at 70°C were observed to test this assumption because after 418 h of heating, the levels of glucose and fructose approached 0, allowing a more rigorous fit of the data to the equations to be tested. Figure 2 shows glucose content as a function of heating time at 70°C . The initial assumption of first order kinetics means that the rate is described as

$$\text{Rate} = dG/dt = kG, \quad [1]$$

where G represents the concentration of glucose and k is the rate constant for the reaction. Rearrangement and integration leads to

$$G = G_0 e^{-kt}, \quad [2]$$

where G_0 is the initial concentration of glucose. As seen in Fig. 2, however, the data do not fit this simple exponential function. When the data are fit using the bi-exponential equation

$$G = G_{0,a} e^{-k_1 t} + G_{0,b} e^{-k_2 t}, \quad [3]$$

with $G_{0,a} + G_{0,b} = G_0$, a large improvement is observed. The fit to a bi-exponential implies that glucose may react by two separate first order pathways, one of which, described by k_1 , is 10 times faster than the other, described by k_2 . A second possibility is that the reaction is not first order.

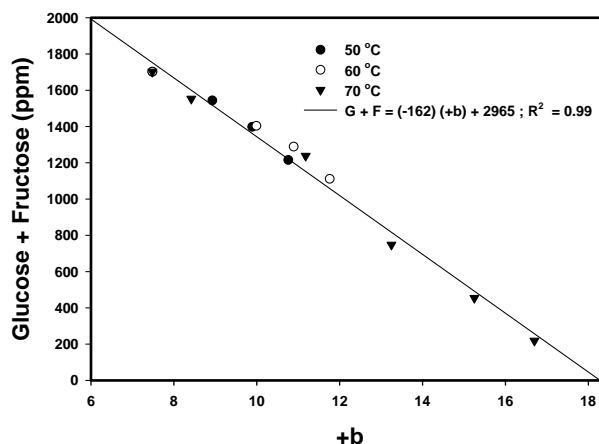


Figure 1. Total reducing sugar concentration as a function of $+b$.

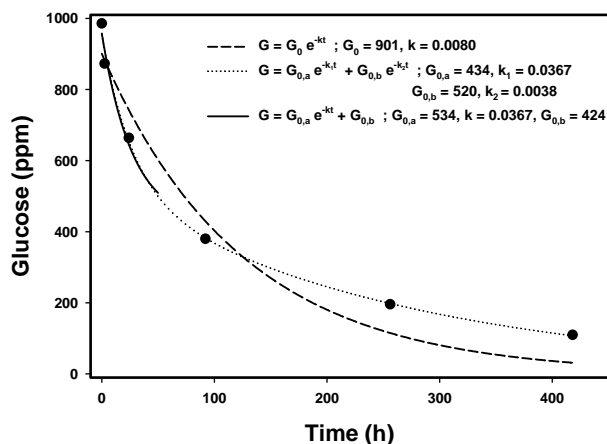


Figure 2. Glucose concentration as a function of heating time at 70°C .

As mentioned previously, determination of reaction orders in solid systems may be exceedingly difficult. To determine the order, and presupposing that $+b$ changes are due to the Maillard reaction, the content of all free amino acids as well as those comprised in available proteins need to be determined as a function of heating time. This is the subject of continuing investigation and lies beyond the scope of the present work, which is to predict $+b$ changes as a result of conventional storage temperatures. To address this issue, the initial assumption was made that over the range of normally encountered cotton storage conditions (temperature and length of time) the first term in Eq. [3] will dominate. That is, the second term plays a significant role only under extraordinarily high temperatures or extraordinarily long storage periods. The validity of this assumption was reexamined subsequent to development of a working model (see below). Under this assumption only the initial reaction period was considered, which

allowed the second exponential term in Eq. [3] to be approximated by a constant. Glucose was monitored to $t_{1/2}$, the time at which $G = 1/2 G_0$, and the results fit using Eq. [4]:

$$G = G_{0,a} e^{-kt} + G_{0,b} \tag{4}$$

The result is seen in Fig. 2, and up to $t = t_{1/2} = 50$ h, Eq. [4] provides a nearly identical fit to the data, with $k(\text{Eq. [4]}) = k_1(\text{Eq. [3]})$. The variables $G_{0,a}$ and $G_{0,b}$ are altered to some degree, however. Fructose exhibited similar behavior (not shown), and the data fit to $t \leq t_{1/2}$ likewise may be fit using Eq. [4], with F replacing G . Application of this treatment to both fructose and glucose at 50, 60, and 70 °C yields the data in Table 3 using Eq. [4]. It is notable that the variables $G_{0,a}$ and $F_{0,a}$ as well as $G_{0,b}$ and $F_{0,b}$ do not vary substantially as a function of temperature. The rate constant k does vary as a function of temperature, increasing in magnitude at higher temperatures. This behavior is consistent with the Arrhenius Equation (Eq. [5]), which states that for many reactions, the rate constant k varies with $T(\text{K})$ according to

$$k = A e^{-E/RT}, \tag{5}$$

where E is the energy of activation for the reaction, R is the gas constant ($= 8.314 \text{ J mol}^{-1} \text{ K}^{-1}$), and A is the pre-exponential factor. Taking the logarithm of each side of Eq. [5] yields

$$\ln(k) = \ln(A) - E/RT. \tag{6}$$

If Eq. [5] is obeyed, $\ln(k)$ plotted as a function of $1/T$ yields a straight line with slope $-E/R$ and intercept $\ln(A)$, allowing E and A to be determined. When the $\ln(k)$ values from Table 3 are plotted vs. $1/T$ for both glucose and fructose, the results, shown in Fig. 3, appear to obey Eq. [5].

Results of $+b$ vs. time at 70 °C are shown in Fig. 4. Similar to glucose and fructose, the results can be fit using a bi-exponential rise to maximum. In this case the equation used is

$$+b = +b_0 + m(1 - e^{-k_1 t}) + n(1 - e^{-k_2 t}), \tag{7}$$

where $+b_0$ is the initial value of $+b$ prior to heat treatment, m and n are variables, and k_1 and k_2 are rate constants. As with glucose and fructose, when the fit is limited to data where $t \leq t_{1/2} = 50$ h, then Eq. [7] may be simplified to

$$+b = +b_0 + m(1 - e^{-kt}). \tag{8}$$

The resulting fit is seen in Fig. 4. Similar treatments for $+b$ vs. t data obtained at 50 and 60 °C yield values for $+b_0$, m , and k at each temperature (Table 4).

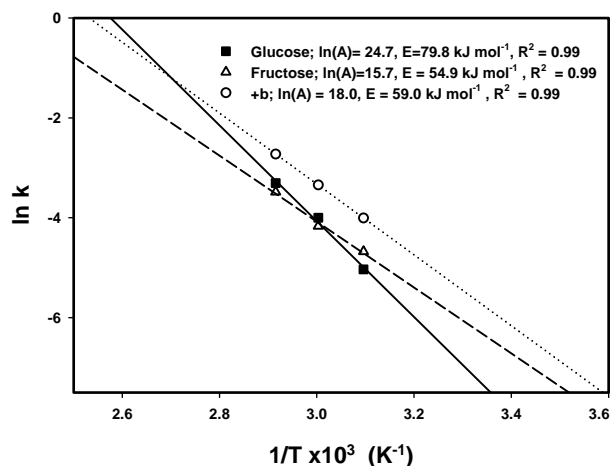


Figure 3. $\ln(k)$ as a function of $1/T$ for glucose, fructose, and $+b$.

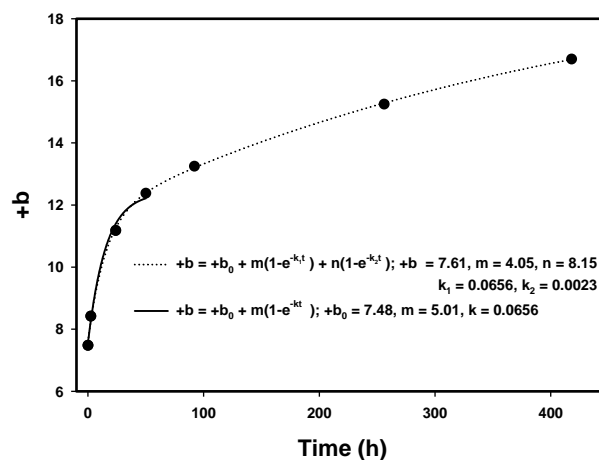


Figure 4. $+b$ as a function of heating time at 70 °C.

Table 3. Regression parameters for glucose and fructose resulting from a fit of concentration vs. heating time using Eq. [4]

Predicted Variable	G _{0,a} (ppm) F _{0,a} (ppm)			k (h ⁻¹)			G _{0,b} (ppm) F _{0,b} (ppm)		
	50 °C	60 °C	70 °C	50 °C	60 °C	70 °C	50 °C	60 °C	70 °C
Glucose	535	528	534	0.0065	0.0183	0.0367	441	452	424
Fructose	210	206	211	0.0093	0.0155	0.0307	502	505	499

Table 4. Regression parameters for +b resulting from a fit of +b vs. heating time using Eq. [8]

Predicted Variable	+b ₀			k (h ⁻¹)			M		
	50 °C	60 °C	70 °C	50 °C	60 °C	70 °C	50 °C	60 °C	70 °C
+b	7.56	7.57	7.48	0.0182	0.0353	0.0656	3.40	4.18	5.01

When $\ln(k)$ is plotted against $1/T$ (Fig. 3), the results indicate that the rate constant for the increase in +b obeys the Arrhenius expression (Eq. [5]). By extrapolation using Eq. [6] and the obtained values of $\ln A = 18.0$ and $E = 59.0 \text{ kJ mol}^{-1}$, the rate constant for the reaction resulting in +b increases may be predicted at any temperature. This allows utilization of Eq. [8] at a given temperature to calculate the rate constant for the change in +b as a function of storage time. Unlike the case for glucose and fructose, however, the variable m from Eq. [8] is not constant as temperature changes. In addition to the rate constant k , a solution for m is also needed. When m is plotted as a function of temperature (Fig. 5), a clear trend is observed. A linear fit with slope 0.0805 and y-axis intercept -22.61 also passes through 281 K ($= 8 \text{ }^\circ\text{C}$) at $m = 0$. Though highly predictable, the cause of this behavior of the variable m is uncertain, but may be a result of decreasing mobility of the reactants as a function of decreasing temperature. Knowing the behavior of k and m as a function of temperature allows prediction of +b at time t at any temperature. To validate this conclusion, 10 g of untreated cotton was allowed to age at 21 °C and 65% RH for 90 d ($= 2160 \text{ h}$). To predict the +b value after this time period, m is first calculated from the linear regression shown in Fig. 5:

$$m = (0.0805)T - 22.61 = 1.0651, \quad [9]$$

where T is in units of K. The rate constant k is next calculated using Eq. [6] and the values for the parameters A and E/R obtained from the linear fit in Fig. 3:

$$\ln(k) = 18.0 - (7096)(1/T) = -6.129 \quad [10]$$

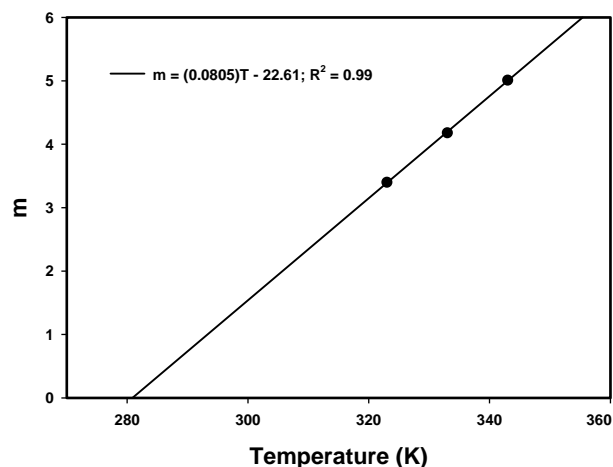
and

$$k = 0.00218 \text{ (} T = 294 \text{ K) .}$$

These values are then substituted into Eq. [8]:

$$+b = +b_0 + m(1 - e^{-kt}) = 8.55.$$

The experimentally determined value for +b using HVI after 2160 h at 21 °C is 8.6 ± 0.7 . This provides considerable substantiation that the model provides a practical method of predicting changes in +b under a large range of storage conditions. As mentioned previously, to arrive at Eq. [8] the assumption was

**Figure 5. Pre-exponential m as a function of T .**

made that the second term in Eq. [7] could be treated as a constant. This is only valid when the cotton is stored for a period less than $t_{1/2}$, the time at which the reducing sugar content reaches 1/2 of its initial value. For longer time periods, +b may change by a greater amount than predicted. Nonetheless, the model predicts the minimum change to +b over a given time at a given temperature. As a hypothetical example, a cotton bale identical to the one treated in this study is purchased in the U.S. and shipped to Asia. The transit time within a cargo container is approximately 21 d ($= 504 \text{ h}$). If the temperature within the cargo container is 30 °C ($= 86 \text{ }^\circ\text{F}$), a reasonable temperature to expect in an unrefrigerated container, then after 21 d

$$+b = 7.5 + [1.78][1 - e^{-(0.0044)(504)}] = 9.08 .$$

It is apparent that the color grade upon arrival in Asia may not match the original value determined in the U.S. just 3 wk prior. Shipping the same cotton via a refrigerated container at 8 °C ($= 46 \text{ }^\circ\text{F}$) results in

$$+b = 7.5 + [0.01][1 - e^{-(0.0007)(504)}] = 7.5 ,$$

or no change in color grade. In Fig. 6, values of +b (calculated using Eqs. [8], [9], and [10]) are shown as a function of storage time at four temperatures encompassed by a range of typically encountered storage temperatures. The practical utility of presenting the data this way lies in the ability to approximate +b changes at any given storage temperature and time. It must be emphasized that the present model relates primarily to the specific cotton used in this study.

Though different cottons will undergo the same chemical reactions leading to +b increases, it is likely that they are dependent upon the levels of reducing sugars present initially, the initial value of +b (+b₀), and possibly the initial pH. A more comprehensive model taking these factors into account is currently in development.

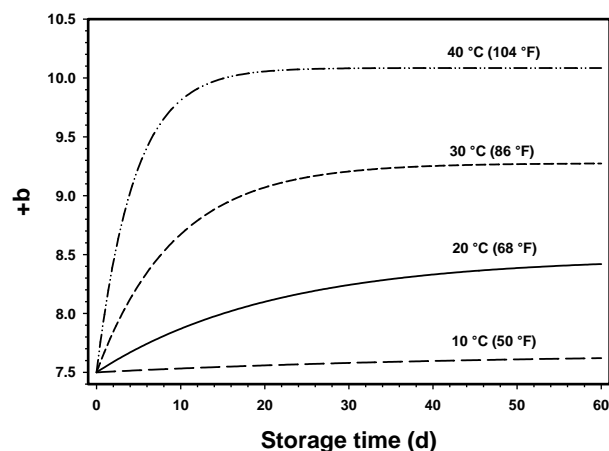


Figure 6. +b as a function of storage time in a range of typically encountered storage temperatures.

CONCLUSIONS

When a bale of cotton was subjected to varying temperature and time conditions, resultant values of reducing sugar content and +b were fit to exponential regressions to give rate constants that are temperature dependent and obey the Arrhenius expression. The general expression for predicting +b is given by Eq. [8], which contains a variable, denoted by m , that is also temperature dependent according to Eq. [9]. A model was constructed that allows prediction of +b as a function of any given storage time and temperature that may be encountered under typical conditions for the particular cotton used in this study. The present model will be the basis for construction of a more extensive model that can be utilized to predict color change in any Upland cotton based on initial +b, reducing sugar content, and pH values.

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