https://doi.org/10.17221/47/2023-RAE

Drying and colour kinetics of decorticated queen pineapple *(Ananas comosus L.)* fibre bleached with a hydrogen peroxide solution

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Citation: De Vela R.J.L., Elepaño A.R. (2024): Drying and colour kinetics of decorticated queen pineapple (Ananas *comosus* L.) fibre bleached with a hydrogen peroxide solution. Res. Agr. Eng., 70: 73–81.

Abstract: The drying and colour kinetics of H_2O_2-bleached pineapple fibres were studied to determine an optimum drying condition and appropriate drying and colour kinetic models. The experiments were conducted under drying air temperatures of 40, 50 and 60 °C, air velocities of 0.27, 0.38 and 0.42 m·s^{-1} and hydrogen peroxide (H_2O_2) concentrations of 1, 3 and 5% by volume arranged in a three-factor factorial experimental design. Colour values were quantified by the CIE*Lab* system where *L** is the lightness value, *a** is redness/greenness and *b** is yellowness/blueness. Total colour difference (Δ*E*), chroma, hue angle, browning index (*BI*) and whiteness index (*WI*) were calculated. The *L** value, *a** value, *b** value, *WI, BI*, hue angle, and chroma were significantly affected by the interaction of the three factors. A non-parametric test was conducted for the drying rate data and showed that the drying rate was influenced by different treatments. Tensile strength was not affected by any of the factors. The optimum drying condition was determined to be 57 °C, at an air velocity of 0.345 m·s^{-1}, and H_2O_2 concentration of 4.8%. The exponential model adequately described drying data. Zero-order kinetic equation described **Δ***E* while *L**, *a*, b**, chroma, hue angle, *WI* and *BI* were satisfactorily described by the first-order kinetic equation.

Keywords: browning index; *CIE*Lab colour system; fiber bleaching; chroma; hue angle; whiteness index

Pineapple leaves yield strong and silky fibres, which are regarded as the finest among the hand-woven fabrics in the Philippines and are considered the queen of Philippine fabrics. Desirable characteristics such as smoothness, glossiness, lightweight, good colour and texture, and high tensile strength, to name a few, account for the wide application of pineapple fibre (Asim et al. 2015).

Piña fibre, as it is popularly known, comes from the leaves of the pineapple plant, which are typically considered waste materials after fruit harvesting. This is derived with the use of either a decorticating machine or through manual scraping. The fibres are then washed with clean or running water and eventually dried under the sun or through the use of mechanical dryers. One of the problems encountered in the process is the unsatisfactory colour of the dried pineapple fibre. Instead of white fibres, greenish fibres are produced, leading to low-quality products.

Drying, being an integral part of pineapple fibre processing shall be understood and properly undertaken to ensure good quality products. In the case of pineapple fibre, where colour and tensile properties are the main quality factors, the drying characteristics of the pineapple fibre must be established

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to attain an optimum drying process with a focus on the abovementioned quality parameters. Process modelling is important in analysing design and optimization of drying to produce high-quality products (Mohammadi et al. 2008). Improvement of the fibre colour as a quality attribute requires knowledge of the colour kinetics involved in the drying process.

In the agricultural processing industry, kinetic parameters such as reaction order, rate constant and activation energy are essential to predict the quality of products. Therefore, kinetic studies are needed in order to minimize undesirable changes and to optimise the quality of a product (Koca et al. 2007). Colour kinetics studies have been done on the processing of several agricultural materials such as dehydrated carrots (Koca et al. 2007), red pepper (Di Scala and Crapiste 2008), green asparagus (Lau et al. 2000), kiwifruit slices (Mohammadi et al. 2008), frozen green beans (Martins and Silva 2002), durian chip (Jamradloedluk et al. 2007), and pineapple puree (Chutintrasri and Noomhorm 2007), to name a few. The majority of these works reported zero-order- or first-order-degradation reaction kinetics.

Hydrogen peroxide (H_2O_2) was used as a bleaching agent in this study to improve the whiteness of the pineapple fibre. It bleaches the fibre by removing lignin, hemicellulose and other surface impurities, thereby improving its brightness (Rayung et al. 2014). This is accomplished by dissociating hydrogen peroxide in alkaline media into perhydroxyl ions (HOO^-), which attack the light-absorbing chromophoric groups of lignin and cellulose (Sundara 1998). When used for bleaching jute, H_2O_2 causes only a small loss of lignin thereby retaining over 70% of the yarn strength (Chatterjee and Pal 1955). H_2O_2 has also been shown to be effective in bleaching knit fabric, fibre composites and cotton textiles (Rayung et al. 2014; Špička and Tavčer 2015; Oliveira et al. 2018).

This study investigated the drying and colour kinetics of decorticated pineapple fibre during hot air drying. Specifically, it aimed to: determine the effects of drying temperature, air velocity and hydrogen peroxide concentration on drying rate, colour values and tensile properties of pineapple fibres; find the optimum combination of drying parameters and bleaching parameters that would yield high drying rate and good quality products; and establish models to describe the drying and colour kinetics during drying of pineapple fibre.

MATERIAL AND METHODS

A three-factor factorial experimental design was employed. The three factors involved were drying temperature (40, 50 and 60°C), air velocity (0.27, 0.38 and 0.46 m·s^{-1}), and hydrogen peroxide concentration (1, 3 and 5% by volume). Three replicates were done for each treatment combination.

The fibre samples (Figure 1) were cut in uniform lengths of approximately 40 cm. Approximately 50 ± 5 g of washed fibres were soaked in a 500 mL H_2O_2 solution for 2 h, maintained at 20 ± 5 °C by placing it in a cold-water bath. The bleached samples were blanched by immersing them in boiling water for five minutes and placing them in a cold-water bath until completely cooled. The drying experiment was conducted in a thin-layer laboratory dryer, which comes with a variable speed blower used to adjust the air flow rate. The heating system consists of four heating elements placed inside a compartment and covered with insulators.

Figure 1. (A) Freshly decorticated pineapple fibre and (B) dried decorticated fibre

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The response variables measured at different treatments were: drying rate, g·min^{-1}, tensile strength and tristimulus CIE*Lab* values (CR-10 Color Reader, Konica Minolta, Japan), while total color difference (ΔE) (Equation 1), chroma (C), (Equation 2), hue angle (h; °) (Equation 3), whiteness index (WI) (Equation 4), browning index (BI) (Equation 5) were calculated (Mohammadi et al. 2008; Saricoban and Yilmaz 2010; Matsuo et al. 2012). In the *CIE*Lab colour system, the L^*, a^*, and b^* values represent the lightness component, the greenness/redness and blueness/yellowness, respectively. The x in Equation 5 describes the relationship of the L^*, a^*, b^* values and how these values influence the browning index. Except for tensile strength, measurements of these parameters were done every 10 min for the first 30 min and every 30 min thereafter.

$$\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{(1/2)} \quad (1)$$

$$C = (a^2 + b^2)^{(1/2)} \quad (2)$$

$$h = \tan^{-1}\left(\frac{b}{a}\right) \quad (3)$$

$$WI = [(100 - L)^2 + a^2 + b^2]^{(1/2)} \quad (4)$$

$$BI = \frac{\left[100 - \left(x - 0.31\right)\right]}{0.17} \quad (5)$$

where: $x = \frac{\left(a + 1.75L\right)}{\left(5.645L + a - 3.012b\right)}$

For tensile testing, dried and brushed pineapple fibre were parallelised and combined in a bundle, 2.54 cm in length and weighing approximately 15–25 mg. Samples were placed in the Instron 4411 Universal Testing Machine (Instron, USA) with 2.54 cm spacing between the clamps and were eventually subjected to tensile loading. The test employed a load capacity of 490 Newtons and was executed at a crosshead speed of 30 mm·min^{-1}. The tensile strength (N·Tex^{-1}) was calculated by dividing the breaking force, N by the linear density, Tex.

Statistical tests were performed using SAS software (version 9.1). The effects of the factors in the response variables were examined by analysis of variance (ANOVA). Response surface methodology (RSM) was employed to optimise the drying operation using the dependent and independent variables.

Experimental data on drying behaviour were fitted to an exponential model, while colour data were fitted to two kinetic models and processed by using Statistica software (version 7). Zero and first-order kinetic rate constants were calculated from non-linear regression. The coefficient of determination (R^2) value was used as the basis in selecting the best fitting for estimation of the parameters of the models.

RESULTS AND DISCUSSIONS

Table 1 summarise the results of the ANOVA performed to test the effect of the factors on the response variables. ANOVA in a three-factor factorial experiment consists of a sequential test of hypothesis, wherein a three-way interaction is tested first before the main effects. Since the interaction effects are significant, one factor can no longer be separated from the rest, so testing the main effects is unnecessary.

Drying rate. The data on drying rate (g·min^{-1}) were not normally distributed and did not have equal variances. Instead of ANOVA, a non-parametric test revealed that there was a significant difference among the treatment combinations in this study. However, in a non-parametric test, the factors that caused the significant difference among the treatment combinations could not be determined.

Lightness value. Except at 1% H_2O_2 concentration, a higher L^* value was attained at lower air velocity and higher H_2O_2 concentration. Higher H_2O_2

Table 1. Summary of the significance of the interaction effects and the mean square values for different parameters

Factors	Final MC	*TS*	*L*	*a*	*b*	*WI*	*BI*	*C*	*h*	ΔE
T × AV	0.012***	0.00007^{ns}	9.933**	1.348*	24.139***	25.927***	62.795***	21.147***	55.03***	20.206*
T × Con	0.023***	0.0004^{ns}	27.308***	6.362***	10.861***	33.696***	25.214***	10.526***	75.525***	28.783**
AV × Con	0.010**	0.0003^{ns}	12.133**	5.341***	15.218***	21.022***	39.24***	14.175***	96.193***	10.272^{ns}
T × AV × Con	0.016**	0.0005^{ns}	44.484***	9.752***	17.789***	53.345***	59.676***	17.098***	172.294***	27.155^{ns}

***,**,* significant at levels of confidence 99, 95 and 90%, respectively; *T* – temperature; *AV* – air velocity; *Con* – concentration; ns – not significant; MC – moisture content (d.b.); *TS* – tensil strenght (N·Tex^{-1}); *L* – lightness; *a* – redness/greenness; *b* – yellowness/blueness; *WI* – whiteness index; *BI* – browning index; *C* – chroma; *h* – hue angle (°); ΔE – total colour difference

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concentration means greater bleaching effect and lower air velocity means lesser exposure to air, causing browning due to oxidation. The slight increase in *L** value at 1% H_2O_2 concentration may be considered insignificant due to its low slope (2.4) compared with that of 3% (8.1) and 5% (7.1) (Figure 2).

Greenness/redness value. Higher *a** values mean that the sample has decreased its greenness and is therefore, approaching the red colour. Fibre samples attained higher values of *a** with increasing temperature (Figure 3A), which means that there was higher degradation of the green pigment in the fibre. Higher drying air temperature means a higher rate of reactions and, therefore, more green pigment degradation.

Fiber samples attained higher *a** values with increasing temperature (Figure 3B) and higher air velocity (Figure 3C). This implied that the degradation reaction also increased with air velocity. Figures 3A and B further illustrate that a greater bleaching effect was attained at higher levels of H_2O_2 concentration.

Yellowness/blueness value. The *b** value, which indicates the yellowness or blueness of the fibre samples, was also significantly affected by the interaction of the three factors. The *b** value increased with increasing temperature at 0.46 $m \cdot s^{-1}$ airflow (Figure 4A). The reversal of effect at 0.27 $m \cdot s^{-1}$ may be considered insignificant due to the low slope of its line (0.06). Lower *b** values were obtained at higher H_2O_2 concentrations (Figures 4B and C). A lower *b** value is desirable since it means closeness to the blue hue in this scale.

Whiteness index. *WI*, a colour parameter which incorporates the *L*, a**, and *b** values were significantly affected by H_2O_2 concentration. Whiter fibres are lighter and are less yellow. A higher whiteness index can be attained at higher levels of H_2O_2 concentration, with temperature and air velocity showing no fixed trend on their effect.

Browning index. *BI* is a parameter associated with the browning phenomenon in the dried product. Although there was a drop in the browning index at 50 °C, Figure 5A shows that at 0.38 and 0.46 $m \cdot s^{-1}$, the browning index generally increased with tempera-

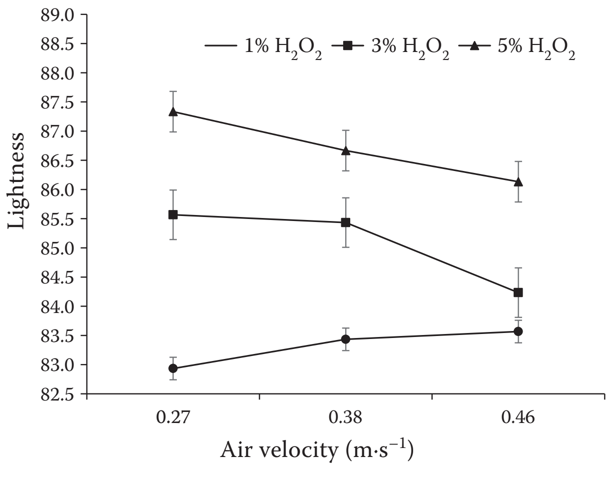

Figure 2. The interaction effect of air velocity and H_2O_2 concentration on the lightness value

Error bars pertain to standard error; H_2O_2 – hydrogen peroxide

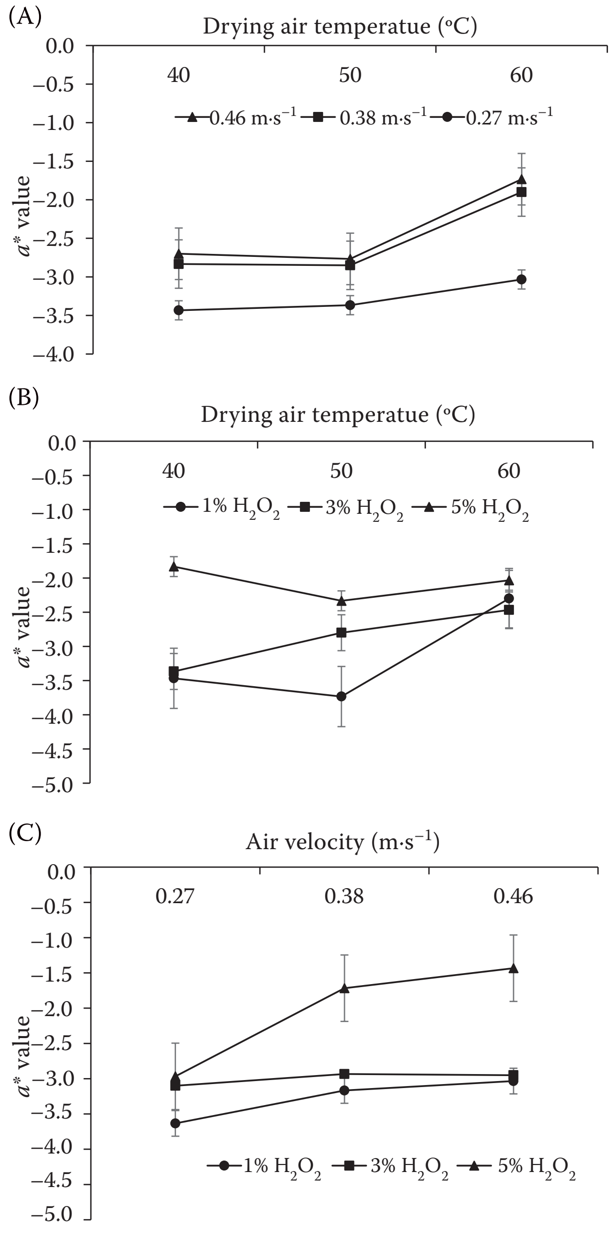

Figure 3. The interaction effect of (A) temperature and air velocity, (B) temperature and H_2O_2 concentration, and (C) air velocity and H2O2 concentration on the final *a** value

Error bars pertain to standard error; H_2O_2 – hydrogen peroxide; *a** – redness/greenness

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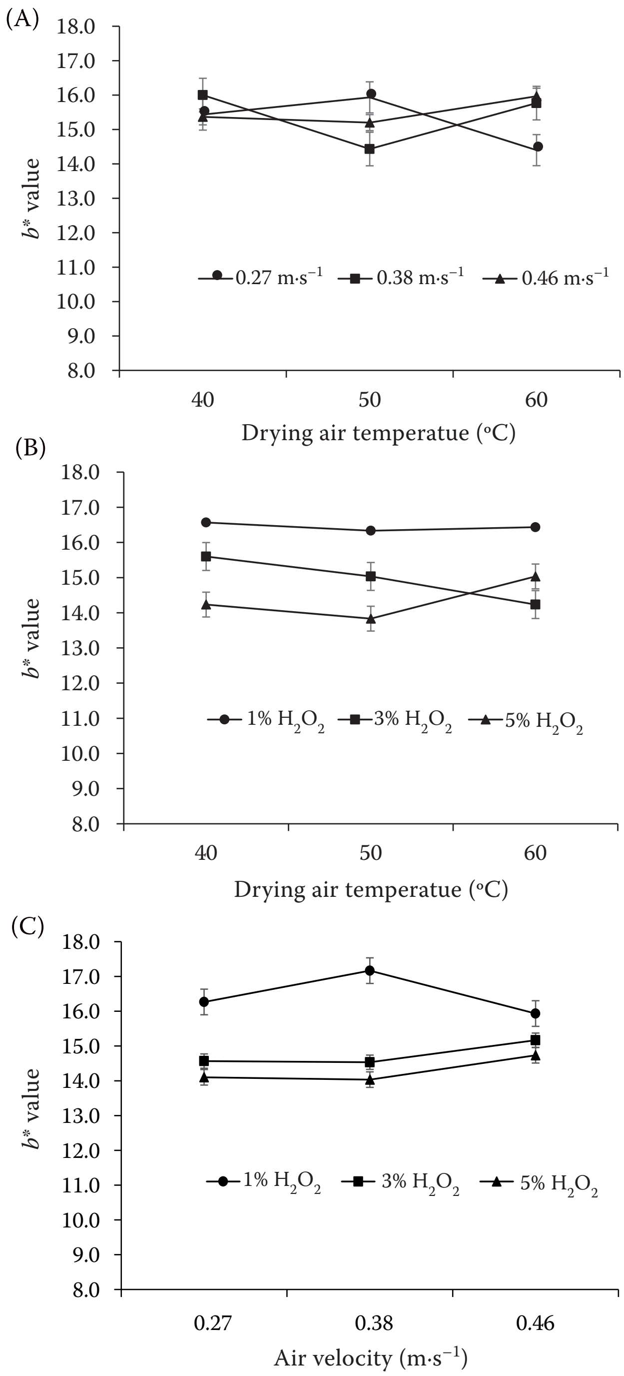

Figure 4. The interaction effect of (A) temperature and air velocity, (B) temperature and H_2O_2 concentration, and (C) air velocity and H_2O_2 concentration on the b^* value

Error bars pertain to standard error; H_2O_2 – hydrogen peroxide; b^* – yellowness/blueness

ture. With higher temperature at 0.27 m·s^{-1}, the product reached the target moisture content at a shorter time allowing lesser exposure to the drying air, hence the reversal of change at this air flow rate. Exposure to air causes browning in most agricultural commodities due to oxidative browning (Cernisev 2010; Ioannou and Ghoul 2013; Mitra et al. 2015).

Figure 5B, on the other hand, shows the effects of air velocity and concentration interaction. At nearly all levels of H_2O_2 concentration, browning index increased with air velocity. Lower browning index implies lesser exposure to air which causes browning reactions. Browning index values were lower at samples bleached with higher levels of H_2O_2.

Hue angle. The Hue angle (°) representing the dominant colour of the fibre samples is also significantly affected by the interaction of the three factors. The greatest increase in hue angle with temperature was observed at a velocity level of 0.46 m·s^{-1}, as indicated by its steep slope (Figure 6A). At a low air velocity level of 0.27 m·s^{-1}, the hue angle exhibited a very slight change with temperature.

For the temperature and H_2O_2 concentration interaction effect (Figure 6B), the trend for the hue angle closely followed that of a^* value. Figure 6C illustrates

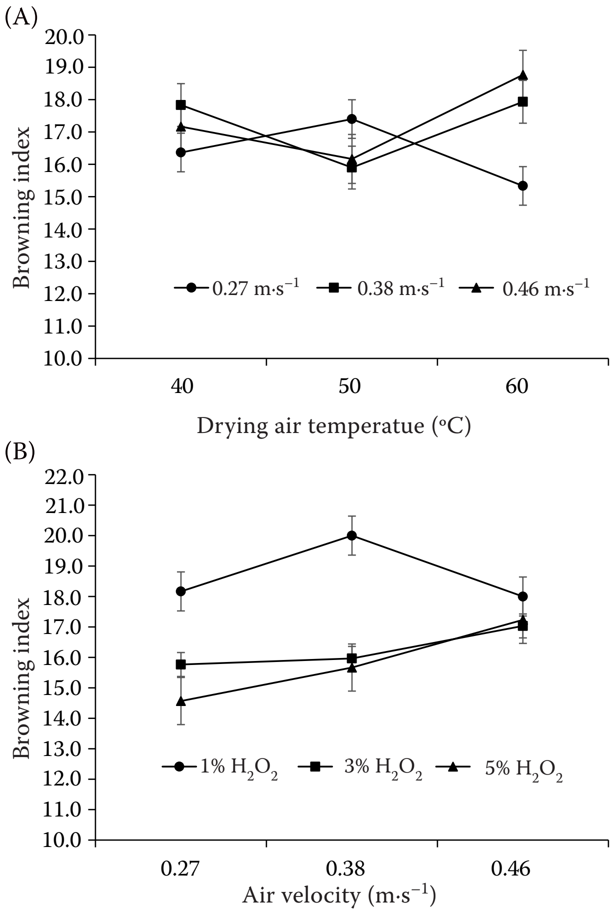

Figure 5. The interaction effect of (A) temperature and air velocity, and (B) air velocity and H_2O_2 concentration on browning index.

Error bars pertain to standard error; H_2O_2 – hydrogen peroxide

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how the hue angle was affected by the interaction of air velocity and H_2O_2 concentration. The Hue angle increased with air velocity. The similarity in the trend observed at the hue angle with that of *a** value implies that a value greatly influences the hue angle of decorticated pineapple fibre.

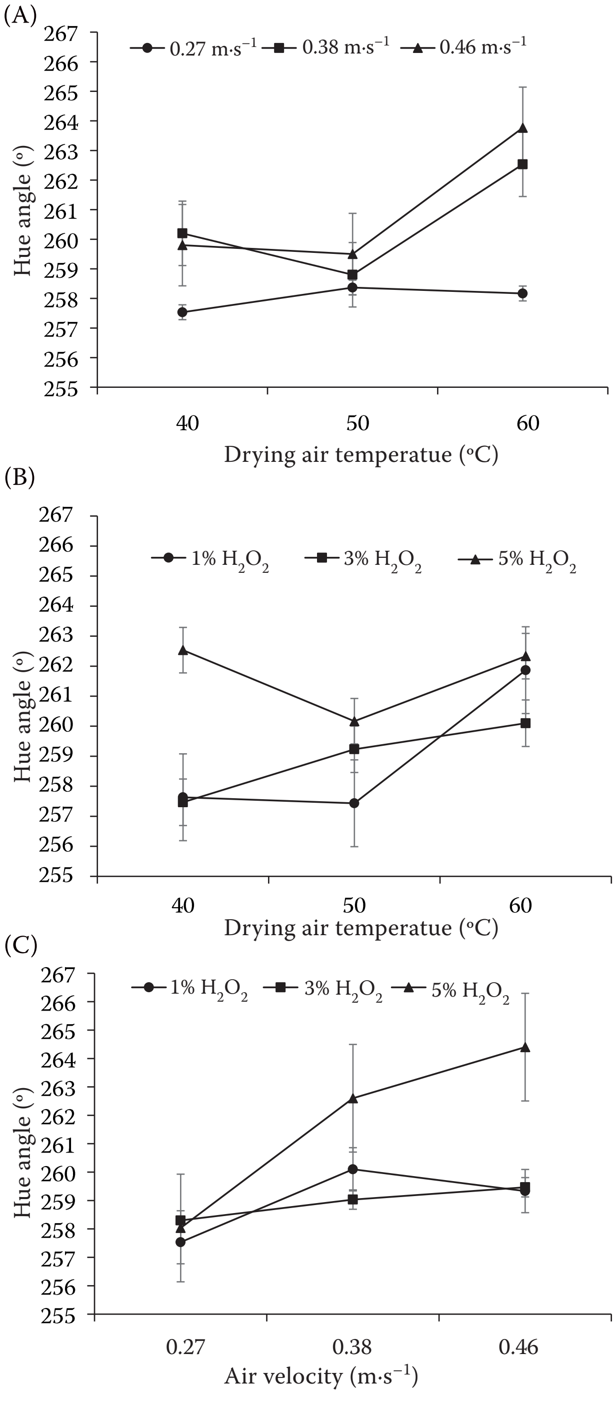

Figure 6. The interaction effect of (A) temperature and air velocity, (B) temperature and H_2O_2 concentration, and (C) air velocity and H_2O_2 concentration on hue angle

Error bars pertain to standard error; H_2O_2 – hydrogen peroxide

Chroma. The three factors significantly affected Chroma, a measure of colour intensity. At low air velocity levels, chroma values decreased with increasing temperature (Figure 7). At air velocity of 0.38 and 0.46 $m \cdot s^{-1}$, chroma increased rather than decreased. Low chroma values are desirable because it means the low intensity of the prevailing colour in the fibre samples. The decreasing trend with temperature at 0.27 $m \cdot s^{-1}$ may be considered insignificant due to its low slope (0.07).

Figure 7B shows that at high levels of H_2O_2 concentration, chroma values were lower. Low chroma values imply dullness or low intensity of the prevailing colour in the pineapple fibre and are more desirable than higher ones.

Tensile strength. The average tensile strength of fibres bleached with H_2O_2 and dried in a hot-air dryer was 22.5 ± 0.2 $N \cdot Tex^{-1}$, which was not signifi-

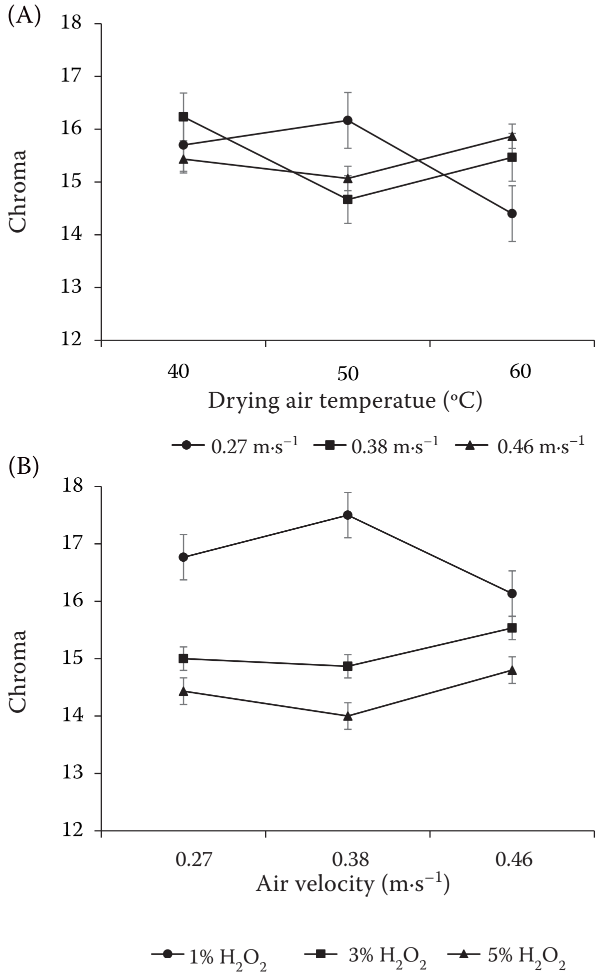

Figure 7. The interaction effect of (A) temperature and air velocity, and (B) air velocity and H_2O_2 concentration on final chroma value

Error bars pertain to standard error; H_2O_2 – hydrogen peroxide

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cantly different from sun-dried, unbleached fibres (23.1 ± 0.2 N·Tex^{-1}). This means that at low levels of temperature and H_2O_2 concentration, the tensile strength was not greatly affected. Theoretically, the tensile strength of the natural fibre is influenced by moisture content. Roselle/sisal polyester-based hybrid composites exhibited a decrease in tensile strength upon exposure to a moist environment (Athijayamani et al. 2009). In this experiment, all the samples tested were in relatively dry conditions or at low moisture content.

Optimum drying conditions. The optimum values of drying temperature (°C), air velocity (m·s^{-1}), and H_2O_2 concentration (% by volume) were evaluated and established using RSM, where contour graphs were constructed for each response variable which was significantly affected by the factors involved in the study. To determine the optimum region in these contour graphs, a desirable range of values was set for each response variable. For the whiteness index, higher values are more desirable since they indicate closeness to the preferred white, while the browning index should be as low as possible. The Hue angle should be as high as possible and close to 270 °, indicating the dominance of the blue hue. Lower chroma values are desirable and indicate low colour intensity of the samples.

Upon examination of the summarised optimum values (Table 2), common ranges of values for independent variables were identified as the optimum drying condition. The optimum drying condition was found to be 57 °C, 0.345 m·s^{-1}, and 4.8% H_2O_2 concentration.

Drying and colour kinetics. Table 3 shows the model developed for each colour parameter. The exponential model can adequately describe the drying behaviour of the decorticated pineapple fibres (R^2 = 0.99). Meanwhile, the L^* value of the fibre samples increased during drying. Most agricultural materials when dried exhibit decreasing L^* values as an indication of browning phenomena (Gupta et al. 2011; Kulwinder and Singh 2014). However, an opposite trend was observed for the pineapple fibre samples. This increase was potentially due to the bleaching and blanching treatments to which the fibre samples were subjected. It should be noted that the increase in the lightness value of the samples decreased at the latter part of drying as indicated by the leveling-off-portion in the graph. This means that the lightness value of the fibres ceases to stop when the product is already dry.

For the redness/greenness scale or a^* value, the values also increased from a more negative value to a less negative one. This means that the pineapple fibre lost its greenness when dried by hot air. A decrease in the b^* value of samples was observed during hot air drying. These changes in a^* and b^* values may be due to the decomposition of the green pigments (Weemaes et al. 1999) and/or non-enzymatic

Table 3. Mathematical models describing moisture and colour changes in pineapple fibre during drying

Parameter	Model	R^2
MR	$MR = 0.996\ e^{-0.016t}$	0.99
L^* value	$L = 79.7\ e^{0.0002t}$	0.92
a^* value	$a = -5.19\ e^{-0.002t}$	0.99
b^* value	$b = 21.7\ e^{-0.002t}$	0.99
WI	$WI = 73.2\ e^{0.0002t}$	0.98
BI	$BI = 28.7\ e^{-0.003t}$	0.99
C	$C = 21.9\ e^{-0.002t}$	0.99
h	$H = 256.9\ e^{0.0002t}$	0.98
ΔE	$\Delta E = 0.179 + (0.15t)$	0.98

MR – moisture ratio; L^* – lightness; a^* – redness/greenness; b^* – yellowness/blueness; *WI* – whiteness index ; *BI* – browning index; *C* – chroma; *h* – hue angle (°); ΔE – total colour difference; R^2 – coefficient of determination; *t* – time; e – 2.718

Table 2. Optimum range of values of the independent variables

Response variables	Optimum range of values		
	drying air temperature (°C)	air velocity (m·s^{-1})	H_2O_2 concentration (% by volume)
Whiteness index	40–60	0.27–0.42	4.8–5.0
Browning index	57–60	0.27–0.345	1.0–5.0
Hue angle (°)	40–60	0.345–0.420	4.8–5.0
Chroma	40–57	0.27–0.4	3.8–5.0
Optimum combination	57	0.345	4.8

H_2O_2 – hydrogen peroxide

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Maillard browning (Saltmarch and Labuza 1982). As a whole, the whiteness index of the samples increased with time due to the increase in L^* value. With an increasing whiteness index comes a decrease in the browning index. Browning phenomena in the samples were basically due to a non-enzymatic browning reaction (Mohammadi et al. 2008). The decrease in the browning index shows that the browning phenomenon in bleached pineapple fibre decreases as the drying proceeds.

Chroma values closely followed that of the b^* values and also decreased as a function of drying time. On the other hand, the hue angle showed very slight changes with the drying time. The hue angle corresponds to whether the sample is red, orange, yellow, green, blue or violet (Mohammadi et al. 2008). As a whole, the total colour difference increased with drying time, similar to what has been reported for the microwave drying and hot air drying of kiwifruit slices (Mohammadi et al. 2008), dehydrated carrots (Koca et al. 2007), red pepper (Di Scala and Crapiste 2008), and pineapple puree (Chutintrasri and Noomhorm 2007).

For the color kinetics of pineapple fiber during hot air drying, zero-order and first-order kinetic models were used. Using Statistica software, the colour data were fitted to zero-order reaction and first-order reaction equations. Based on the R^2 value, the best model to describe the kinetics of colour change for every parameter was selected. Zero order kinetic equation adequately described the change in total colour difference with R^2 ranging from 0.73–0.97.

Other colour parameters such as L^*, a^*, b^*, h, C, WI, and BI were satisfactorily described by the first-order kinetic equation. Other studies, such as those conducted for pineapple puree (Chutintrasri and Noomhorm 2007), kiwifruit slices (Mohammadi et al. 2008), and bamboo shoots (Bal et al. 2011) have described a value using zero-order kinetic equation. For pineapple fibre, both zero and first-order kinetic equations have a relatively high R^2 value of 0.92 and 0.93, respectively. The first-order kinetic study was chosen as a superior fit in this study.

First order kinetic equation describing the change in b^* value (average R^2 = 0.92) with time was consistent with other studies, pineapple puree (Chutintrasri and Noomhorm, 2007), kiwifruit slices (Mohammadi et al. 2008), peach puree (Ávila and Silva 1999) and bamboo shoot (Bal et al. 2011). The same was true for both chroma (average R^2 = 0.92) and browning index (average R^2=0.89).

CONCLUSION

The L^*, a^* and b^* values, whiteness index (WI), browning index (BI), hue angle (h), and chroma (C) were significantly affected by the interaction of the three factors involved in the study. Total colour difference was influenced by the interaction of temperature and H_2O_2 concentration only. The drying rate was affected by the different treatments while tensile strength was not significantly affected by any of the factors. The optimum drying condition was determined to be 57 °C, 0.345 $m \cdot s^{-1}$ air velocity, and 4.8% H_2O_2 concentration.

The exponential model adequately described the drying data, while the zero-order kinetic equation adequately described the total colour difference. Other colour parameters such as L^*, a^*, b^* , h, C, WI, and BI were satisfactorily described by the first-order kinetic equation.

The study employed low levels of temperature and H_2O_2 concentrations, which did not affect the tensile strength of the dried fibres. Findings show that the use of H_2O_2 as a bleaching agent for pineapple fibres can practically and safely be employed by pineapple fibre processors. Use of higher levels of H_2O_2 concentration is not recommended for economic reasons. However, the effect of the maturity of the leaves on tensile strength can be investigated. Other varieties of pineapple fibre and other cellulosic fibres like hemp, linen, cotton, ramie and sisal can also be subjected to this kind of drying and colour kinetic study. To see the possibility of exceeding the colour quality of sun-dried pineapple fibres, a preliminary experiment on using ultraviolet (UV) light during drying can be performed.

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Received: May 15, 2023
Accepted: December 13, 2023
Published online: June 23, 2024