## The Effect of Temperature on Thermochromic Leuco Dyes: A Meta-Analysis

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Abstract. The effect of temperature treatment, either increasing or decreasing the temperature of leuco dyes, can produce thermochromic by looking at the changing color of samples resulting from experiments. The colors that are seen namely brightness (L), a\* value, b\* value, chroma, degree of hue (°Hue), color difference ( $\Delta E$ ), and color density (CD). This research was conducted to analyze the temperature treatment of the thermochromic leuco dye against color characteristics resulting from meta-analysis using secondary data from pre-existing research. The total number of journals used was 21 studies and the data obtained were 82 sets of treatments. The data were then analyzed using IBM SPSS software, which used quadratic and linear regression models. The results of the study with a temperature range between -8°C to 95°C show that the effect of temperature treatment with an increase in temperature can cause a significant elevation impact on brightness values (p <0.002, R<sup>2</sup> 0.174), b\* (P <0.01, R<sup>2</sup> 0.146), color difference ( $\Delta E$ ) (P <0.0012, R<sup>2</sup> 0.117), and °hue (P <0.053, R<sup>2</sup> 0.087). A significant reduction effect can be seen in the chroma value (P <0.007 R<sup>2</sup> 0.134) and the color density (P <0.007, R<sup>2</sup> 0.128). The impact of temperature treatment was not significant (p > 0.1) for a\* value.

Keywords: Color characteristic; crystal violet lactone (CVL); leuco dyes; meta-analysis thermochromic

#### 1. Introduction

Intelligent packaging can provide information and a communication function for consumers based on its ability to detect, record, track, and sense changes that occur in a product. Intelligent packaging implementations are widely used as sensors to review the quality or condition of a packaged product (Putri *et al.*, 2019a; Putri *et al.*, 2019b; Warsiki & Rofifah, 2018; Rosalina *et al.*, 2022; Rostini *et al.*, 2023). One example of intelligent packaging implementation as a temperature sensor is thermochromic ink and labels (Nofrida *et al.*, 2013; Warsiki *et al.*, 2013; Warsiki *et al.*, 2018). In the thermochromic, color is the main parameter because thermochromic is used as a sensor to detect temperature changes during storage by changing the color of the label/package (Nofrida *et al.*, 2013; Chen *et al.*, 2019). Several important color parameters that can be used as an indicator for proper thermochromic function and change its color with temperature changes are brightness or lightness (L value), a\*value, b\* value, chroma, degree of hue (°Hue), color difference (AE), and color density (CD). With these parameters, the

strength of a color can be measured, and it can be seen how much the temperature influences the color value. Color will undergo a reversible thermochromic process with temperature variations (Baron & Elie, 2003). The color changes are due to structural modification as a function of temperature (Fitria *et al.*, 2017).

Several studies regarding thermochromic using leuco dyes have been conducted (Panák et al., 2015; Bourque, 2014; Bourque & White, 2015). Each author described several findings on the thermochromic effect of leuco dyes that are widely used, namely, Crystal Violet Lactone (CVL), in different temperature treatments so that it can produce color changes. Unfortunately, their research did not explain how contrast results from the color value, and further, the color is only seen visually, so further researchis needed to make a conclusion objective from temperature treatment such as using meta-analysis. Many studies have used meta-analysis (Irawan et al., 2021; Sjofjan et al., 2021; Prihambodo et al., 2021). Apart from being widely used in the medical field, meta-analysis is also widely used in the field of animal husbandry, such as the one that was done by Jayanegara and Palupi (2010) to measure the effect of various kinds of food tannin concentrations on digestion of ruminants. Thus, this study was conducted to determine the effect of temperature treatment on the thermochromic leuco dyes on the obtained color characteristics at various temperatures using quantitative analysis of the regression model, namely by metaanalysis of various treatments from the obtained empirical data. The specific objectives of this study were to obtain tabulated data from selected journals, to obtain numerical data from plot images and graphs and analyze them into their color values, and then to figure out how significant or un-significant the effect of temperature increase on color properties.

#### 2. Methods

The materials used in this research were various published articles and reports, including international journals, theses, and literature from the internet with a reputation. The steps were then (i) collecting data/journal/scientific paper; (ii) selecting them in related fields; (iii) extracting data and (iv) analyzing statistically.

### 2.1. Collecting data/journals/scientific papers

The research methodology employed in this study involved gathering a diverse range of published sources such as articles, journals, and theses. A quantitative approach was adopted for data analysis. The data and information in this study were obtained through secondary data, including internationally recognized journals ranked from Q1 to Q3, reputable theses, and literature from credible online sources. These sources were accessed through reputable academic databases such as Google Scholar, Scopus, and Science Direct. It is important to note that the journals and theses utilized in this research predominantly consisted of studies conducted directly

in laboratory settings, thereby containing primary data to support the analysis.

# 2.2. Selection of journals and scientific papers

The number of collected journals regarding thermochromics and their implementation in the packaging sector was from the year of 1998 to 2023. Journals were selected using Mendeley Desktop to read abstracts and find keywords more easily. The keywords used for reference searches were thermochromic, leuco dye, reversible, color-change, and crystal violet lactone. The keywords were selected based on their significance in the field and their potential to capture relevant literature. Specifically, the research focuses on organic thermochromic obtained from leuco dyes, and the research also showed reversible thermochromic properties, which indicate changes in temperature along with the color change mechanism. The study described its results, namely, observation of the material characteristics used in the form of color value changes that occur along with the changing temperatures. The total number of journals that ultimately matched all the keywords were processed and entered into the database. All data collected were tabulated in Excel with equalized units.

In this study, the integration of quantitative data, which relates to temperature and color characteristics, used the quadratic and linear regression methods. Regression analysis was performed using the IBM SPSS 23 software version. The analysis was performed by filling in the variables and then entering the tabulated data in Microsoft Excel. After that, the data entered were analyzed using Curve Estimation to see the comparison of the analysis results linearly and quadratically. If the results of the quadratic analysis show p > 0.05 (insignificant), it was necessary to be continued linearly.

# 2.3. Process of Extracting, Converting, and Calculating ColorValues

Many of the journals acquired lacked raw numerical data; instead, they predominantly presented data in graphical or image formats. Consequently, to extract precise numerical values from these graphs, a meticulous process was undertaken. This effort involved plotting the graphs using WebPlotDigitizer software to ensure accurate data extraction and analysis. Therefore, data in these graphs need to be extracted by plotting them out to obtain precise accuracy using WebPlotDigitizer software. The graph was stored in the form of an image and then it was entered into this software to determine the X and Y axes. Then, the data plot was taken out and tabulated in Microsoft Excel. The data from the research results, which were in the form of images, were taken from the image by using an imagecolorpicker until it produced RGB color values. Then, it was converted into XYZ and L\* and b\* values using a color conversion calculator with EasyRGB software.

# 2.4. Linear and Quadratic Regression Analysis

In this study, the integration of quantitative data, which relates to temperature and color characteristics used the quadratic and linear regression methods. Regression analysis was processed using the IBM SPSS 23 software version. The analysis was performed by filling in the variables first, then entering the tabulated data in Microsoft Excel. After that, the data, that have been entered, were analyzed using Curve Estimation to see the comparison of the analysis results linearly and quadratically. If the results of the quadratic analysis show p > 0.05 (insignificant), it was necessary to be continued linearly.

## 3. Results and Discussion

#### **3.1.** Collecting Data

The total selected journals were 21 studies between the periods of 1998 to 2023. The amount of data used to study the effect of temperature treatment on the thermochromic leuco dyes was 82 data sets. The temperature ranged from - 8°C to 95°C, with different measurements on each journal. The journals used in this research can be seen in Table 1. The statistically processed data that had been collected and obtained from those journals revealed the descriptive statistical results as presented in Table 2.

Studies	Reference	Year	Temperature (°C)	Developer	Solvent
1	Zhang <i>et al</i> .	2017	25-55	Bisphenol A	Hexadecanol
2	Panák <i>et al</i> .	2015	10-50	Bisphenol A	Tetradecanol
3	Bourque	2014	25-70	-	
4	Bourque & White	2015	25-95	Octyl Gallate	Octadecanol
5	Panák et al.	2015	25	Propyl Gallate	Tetradecanol Hexadecanol
6	Panák <i>et al</i> .	2017	25-50	Bisphenol A	1-Tetradecanol
7	Kulcar et al.	2010	15-45	Binder	Ink
8	Kulcar et al.	2012	25-60	Binder	Ink
9	Kulcar et al.	2009	25-40	Binder	Ink
10	Seeboth et al.	2007	25-40	Bisphenol A	1-Tetradecanol
11	Bašnec et al.	2016	-8-8	Benzyl-4-hydroxy benzoate	Methyl Laurate
12	Burkinshaw	1998	65	Bisphenol A	Stearic Acid
13	Bašnec et al.	2016	5-25	Benzyl-4-hydroxy benzoate	1-dodecanol
14	Jin <i>et al</i> .	2017	25-40	Bisphenol A	-
15	Malherbe et al.	2010	3-25	Bisphenol A	1-dodecanol
16	Raditoiu et al.	2016	25-50	Phenolphthalein	1-Tetradecanol
17	Chowdhury et al.	2013	20-28	Binder	Ink
18	Zhang <i>et al</i> .	2019	5-35	<b>Bisphenol</b> A	-
19	Geng et al.	2018	15-40	Bisphenol A	1-Tetradecanol
20	Tozum et al.	2018	25-40	<b>Bisphenol</b> A	1-Tetradecanol
21	Jakovljević et al.	2017	12-40	Binder	Ink

Table 1. The compiled data used for meta-analysis.

The effect of increasing temperature treatment caused a significant elevation in brightness values (p <0.002, R<sup>2</sup> 0.174), b\* (P <0.01, R<sup>2</sup> 0.146), color differences (P <0.0012, R<sup>2</sup> 0.117) and the degree of hue (P <0.053, R<sup>2</sup> 0.330). A significant reduction effect can be seen in the chroma value (P <0.007, R<sup>2</sup> 0.134) and the color density (P <0.007, R<sup>2</sup> 0.128). The impact of temperature treatment was not significant (p> 0.1), which was only at the a\* value. The regression equations with both linear and quadratic models for the effect of temperature treatment on color characteristics can be seen in Table 3.

Parameter	n <sup>a</sup>	Mean	SD	Min	Max
Temperature T (°C)	82	32.62	17.37	-8.00	95.00
Colorimetric					
properties	70	50.63	27.29	5.36	100.00
Lightness					
a*	60	7.34	16.31	-36.91	58.50
b*	60	-20.47	22.87	-79.74	24.70
Chroma	72	28.37	22.83	0.00	98.89
Color difference ( $\Delta E$ )	73	62.99	36.05	0.93	170.95
oHue	40	289.18	22.84	242.71	360
Color density	75	0.85	0.58	0.00	2.23

Table 2. The statistical description of the effect of temperature on color properties

A total of 82 treatment data; n: number of observations; SD: standard deviation; Min: minimum; Max: maximum

Table 3. Linear and quadratic regression for the	ne effect of temperature treatment	on color properties
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Parameters	Model	Variable	Intercept	Slope	P value <sup>c</sup>	R2
Colorimetric						
properties Lightness	0	Т	37.94	0.11	**	0.174
Lightness	Q	т Т <sup>2</sup>	57.74	0.01		0.174
a*	Q	T	13.25	-0.20	NS	0.029
		T <sup>2</sup>		0.001		
	L	Т	12.50	-0.15	NS	0.028
b*	Q	Т	-3.38	0.59	**	0.146
		T <sup>2</sup>		-0.001		
Chroma	Q	Т	42.78	-0.39	**	0.134
		T <sup>2</sup>		-0.001		
Color difference $(\Box E)$	Q	Т	42.33	0.57	*	0.117
· · ·		т2		0.002		
Degree of hue (°H)	Q	Т	294.37	0.853	**	0.330
,	-	т2		0.016		
Color Density	Q	Т	1.09	-0.002	**	0.128
-		T <sup>2</sup>		0.00		

<sup>c</sup> This model is significant (\*\*) at  $P \le 0.01$ , significant (\*) at  $P \le 0.05$ , tends to be significant (\*\*\*) at  $P \le 0.10$ , not significant (NS) at  $P \ge 0.10$ 

The results from Table 3 showed that the parameter values of brightness, b\*, chroma, color difference, degree of hue, and color density fit in quadratic model analysis; in contrast, the

parameter values of a\* were fit in linear and quadratic models. The biggest  $R^2$  value was 0.330 for the degree of hue parameter, which means that temperature treatment affected 33% of the change in °Hue. This value showed that there were other factors influencing the °Hue change. According to Burkinshaw *et al.* (1998), color precursors, which are included in the acid and solvent groups, become more intense and contrast so that they can change the position of the color. This change can then make the color produced as a sensor for thermochromic. Additionally, the various kinds of leuco dyes can also affect the resulting color parameters. The value of  $R^2$  obtained from each parameter tended to be small, which was acceptable in a meta-analysis test. This result was because the data obtained and used in the test ware varied and cannot be subjective.

#### **3.2.** Color Brightness

The value of L or Lightness indicates the brightness of a color sample. The value of L represents the characteristic or color properties of the reflected light, which results in a value ranging from 0 to 100. The lower the value, the darker the color that the sample will produce, while the higher the value, the brighter the color of the sample will be. Color changes occurring in color pigments are caused by modification or the influence of increasing or reducing temperatures. For example, the environment and treatment of the sample, when the temperature rises, can make discolorations or changes from colored to colorless (Zhang *et al.*, 2019). Changes in the lactone ring structure of the leuco dye can explain these changes. An open lactone ring will produce a denser color and more contrast, resulting in a solid (solidified) transition. Meanwhile, when the temperature increases, the structure in the lactone ring will close, and the transition turns into liquid so that the color will fade until it becomes colorless, as shown in Figure 1.

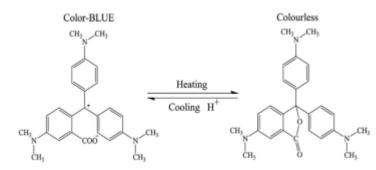


Figure 1. Leuco dye structure with open ring (left) and closed ring (right) (Panák *et al.*, 2015)

In this research, the maximum L value would be obtained when the temperature reaches 95°C, in which this value will reach 100 (Bourque & White, 2015). The minimum value of L was at about 5.36. The largest L value from other studies also reached 90, but did not reach high temperatures up to 95°C, the maximum was only 45°C. It can be caused by various factors from

the treatment and other factors from the thermochromic components used on both color precursors and solvents. The higher the temperature, the hotter the response is to be received, so that the dye will be able to emit energy, one of which is light, and then the dye becomes brighter. From the data collected in this study from various previous research, it was shown that increasing the brightness of the sample due to increasing temperature tends to follow the quadratic regression equation, as Figure 2. It can be said that the effect of increasing temperature on changing the brightness of the sample was significant in P <0.01. This is a relatively low R<sup>2</sup> value, suggesting that the model does not explain a large portion of the variance in the lightness data, and there may be other factors affecting lightness that are not accounted for by temperature alone.

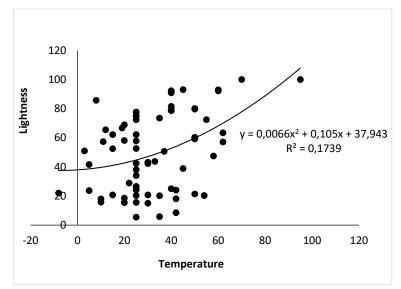


Figure 2. Quadratic regression graph of brightness

Kulčar *et al.* (2010) also described similar phenomena, changing brightness was a result of the temperature changes. The value of L can form a circle on the graph, which serves to determine the stability of the thermochromic as a sensor that changes the color reversibly as the temperature changes (Jakovljević *et al.*, 2017). The higher the temperature, the higher the rate of changein L value. According to Fitria *et al.* (2017), the increase in the value of L was caused by the degradation process of natural dyes due to the effect of temperature. Furthermore, the pheophytin reaction, namely the release of the Mg2 + ion on chlorophyll compounds and substituted by H + ions cause the green of the chlorophyll to turn brown (Jakovljević *et al.*, 2017). In some cases, this thermochromic color change is easy to observe by the naked eye and it often occurs over a small temperature interval.

# 3.3. Chromatic Coordinate Value of a \* and b \*

The CIE Lab color scale is uniform and it describes all colors visible to the human eye. In a uniform color scale, the differences between plot points in the color space can be equated to see the differences in the color Fitria et al. (2017). In this research, the result of a\* value was not significantly different with p > 0.1 and it had a negative relationship with temperature increase. Additionally, the result of b\* value increased significantly (p <0.01). At low temperatures, the b\* value was more positive, which indicates a strong degree for the red, while the b\* value was more negative indicates a strong degree for the blue. The increase in the value of b\* was more noticeable than that of a\*. This phenomenon is because the leuco Crystal Violet Lactone (CVL) dyes produce a blue-purple color when it is at low temperatures. CVL will turn colorless when the temperature is high. Research of Burkinshaw (1998) and Panák et al. (2015) explained that the purple-blue color, which was the maincolor of CVL, was formed due to the lactone ring that was opened. In this phase, the CVL would increase its polarity and hydrogen-bonding ability of the color precursors (phenolic acid), including that which interacts with solvents, in the solid transition. However, this research showed that other factors greatly affect the change in the a\* value, namely, the acids from the color precursor, such as solvents, which are used to interact with other components to form thermochromic. Additionally, from the results of the hue degree value (°H), the maximum of this value was in the red-colored area. Thus, the higher the area temperature of the experiment, it would have resulted in the redder color range of the CVL.

On the other hand, the increase in the value of b\* was proportionally aligned to the effect of increasing thetemperature of the treatment. An increase in the value of b\* indicates that the results of blue were fading away, while the decreasing value of b\* indicated the optimum degree for blue. The parameter value of b\* can be used as a parameter to determine the thermochromic presence of CVL leuco dyes, and it indicates a color transition. The blue refers to conditions that leuco dye is below room temperature. In such commercials applied to certain food packaging such as cold drinks, it can state that the product is ready for consumption (Abdellaoui *et al.*, 2020).

# 3.4. Degrees of Hue (°H) and Chromaticity

The value of the degree of hue and chroma are both color properties that show color visually, which is obtained from calculating the a\* and b\* thus making it makes easier to identify the color change occurs. The degree of hue represents the actual degree of color and it is divided into red, yellow- green, blue, and purple. The degrees of hue are also differentiated to determine the redness and greenness of the light and it is associated with wave lengths of light (Fitria *et al.*, 2017). The results of the analysis show that the minimum degree of hue has a degree of about 242.71 that according to CIELAB colorspace, it is in the blue area, while the maximum value has a degree of 360, which is in the purplish-red area. A researcher (Suryantari & Flaviana, 2017) stated that an increase in temperature will increase the value of the <sup>o</sup>hue and it indicate a color

change. The higher the storage temperature, the higher the hue degree value and it indicates the greater color change. Table 4 explains the range of color that resulted from the value of <sup>o</sup>Hue.

The chroma determines the color purity and color saturation resulting from a dense range to nearly white. The higher the chroma value, the higher the color saturation will be (Warsiki *et al.*, 2019; Putri *et al.*, 2019a). Results of the analysis using quadratic regression in Figure 3 show p <0.01, which means it was significant in chroma value for research done previously, butit has a negative relationship with increasing temperature.

Degree of hue value (°Hue)	Color range area
342° - 18°	Red-purple
18º - 54º	Red
$54^{o} - 90^{o}$	Yellow-red
90°-126°	Yellow
126 ° - 162°	Yellow-green
162° - 198°	Green
198° - 234°	Blue-green
234° - 270°	Blue
270° - 306°	Blue-purple
306° - 342°	Purple

Table 4. The range of colors that resulted from <sup>o</sup> hue (Francis, 1995)
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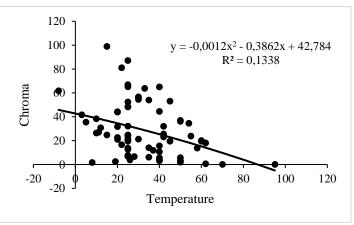


Figure 3. Chroma quadratic model regression graph

The chroma value is identical to the density of a color, which is also determined by the color's brightness. According Durasevic (2016), the chroma value decreases because the lactone rings in the leuco dye will be closed due to the impact of heat and temperature increase. The hydrogen bonds, the result of interactions with the color precursor and its embedded with dye structure, will be released. In the case of LVC, color change occurred in the transition from solid into liquid as a result of interactions between solvents and color developers due to temperature increase.

However, cooling treatment will transform the liquid back into solid. The intensity of the color contrast was associated with the ratio of the lactone ring opened (solid phase) and closed (liquid phase) (Panák *et al.*, 2017).

## **3.5.** Color Difference ( $\Delta E$ )

The value of the color difference or the total color difference is a parameter of the overall color change. The color difference is usually denoted by  $\Delta E$ . The increase in  $\Delta E$  value indicates a big enough color change along with the temperature change. Also, a larger  $\Delta E$  value can indicate a decrease in color intensity from the original color. Accordingto Zhang *et al.* (2019), through the thermal cycle, the  $\Delta E$  value can indicate the stability of the leuco dye in providing a thermochromic effect in a measured temperature range as well as the color changes. Based on the results of the analysis with the quadratic model, it shows a significant relationship between the temperature rise and the color difference (P <0.05). This was in accordance with the research conducted by Nofrida *et al.* (2013) and Fitria *et al.* (2017) that proves a proportionally aligned relationship between  $\Delta E$  and temperature. The higher the temperature, the higher the resulting  $\Delta E$  value will be. The average value of the color difference from each data was based on the table presented in Table 5 (Fu & Hu, 2017).

Color differences (∆E)	Visual Perception	Color variations
0-0.5	Trace	Less obvious color change
0.5-1.5	Slight	Slightly change
1.5-3.0	Noticeable	Change can be seen clearly
3.0-6.0	Appreciable	Marked change
6.0-12.0	Strong	A very marked change
> 12.0	Very strong	Change to another color

Table 5. The relationship between color differences and visual perception

# 3.6. Color Density

Color density represents the ability of a surface or material to absorb light. Generally, the color density is also used to measure the thickness of the dye coating. CVL thermochromic leuco dye is not only in the form of pigment powder but also in the form of inks and pigments that are dissolved in certain solvents. This makes the color density measurement more specific. According to Sudhakar (2008), organic thermochromic such as leuco dyes include dyes that have high color density, can be used in a wider variety of applications. Color density can indicate the ability of a chemical compound to interact in forming colors at a certain value, such as the ability of color

precursors to help produce more contrasting colors and solvents with chain bonds that can affect temperature and solid or liquid transitions to produce a specific color value (Bourque & White, 2015).

Based on the results of the analysis with the quadratic model, p value <0.01 means that there was a significant relationship, although it was a negative relationship. The effect of increasing the temperature makes the color density and the value decrease. The higher the temperature, the more its color density  $\leq 0.1$  and it will be classified as colorless. At room temperature and below room temperature, the resulting density is in the range of 0.5 to 2 so according to the density table (Table 6), it shows that at room temperature and below, it will produce colors that tend to be dark.

<b>Color markers</b>	Color density (CD)
Colorless	$CD \le 0.1$
Bright	0.1 < CD < 0.5
Medium	$0.5 \le \mathrm{CD} < 0.8$
Dark	$0.8 \le CD$

Table 6. Thermochromic sample color marking as color density (Bourque & White, 2015)

## 4. Conclusion

The significant change in color properties due to temperature treatment of thermochromic leuco dye was deduced from an array of data sets sourced from previous studies. This insight is consistent with findings from meta-analysis studies, further affirming the observed effect. Data analysis performed by linear and quadratic regression models concludes that the parameters of brightness (L), b\* value, the degree of hue ( $^{\circ}$ Hue), chroma, color differences ( $\Delta E$ ), and color density (CD) follow the quadratic regression models, while the a\* value parameter complies with linear regression models. The color parameter showed a significant increase as the temperature increased in brightness, b\* value, and the degree of hue. The significant reduction was in chroma and color density, while the a\* value was not affected significantly. The temperature elevation made the values of several color properties, namely, brightness, b\* value, color difference, and degree of hue higher. Meanwhile, the chroma parameter and color density decreased along with an increasing temperature of the experiment treatment. Based on the research findings, it is recommended to consider the impact of temperature on thermochromic leuco dyes when designing and utilizing such materials. Specifically, attention should be paid to the fluctuation of color properties, such as brightness, b\* value, degree of hue, chroma, and color density, in response to temperature variations. This understanding can inform the development of more accurate and reliable thermochromic systems for applications where precise color control is essential, such as in packaging, sensing, or visual indicators. Additionally, further research may be warranted to

explore the underlying mechanisms driving these temperature-induced changes in color properties,

facilitating the optimization of thermochromic materials for diverse practical uses.

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