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Ageing Behavior of Temperature Sensitive Leathers

Sıcaklığa Duyarlı Derilerin Yaşlanma Davranışları

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AGEING BEHAVIOR OF TEMPERATURE SENSITIVE LEATHERS

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ABSTRACT: Enhancing fastness properties of thermochromic dyes and pigments provide unique and assertive design opportunities and an innovative approach for leather industry. For this purpose, the ageing behavior of thermochromic pigments (cold and touch activated) used in leather finishing process by the addition of UV absorbent and cross-linker was investigated. The color changing effect of thermochromic leathers was determined spectrophotometrically prior and subsequent to accelerated ageing tests performed under two different conditions (only heat and heat associated with relative humidity). No significant effect of ageing was found on thermochromic leathers. However, a partial decrease was determined for the cold activated leathers and it was prevented by the use of UV absorbent and cross-linker.

Keywords: Accelerated ageing, thermochromic leathers, color changing effect, color measurement

SICAKLIĞA DUYARLI DERİLERİN YAŞLANMA DAVRANIŞLARI

ÖZET: Termokromik boyaların ve pigmentlerin haslık özelliklerini artırmak deri endüstrisine benzersiz ve iddialı tasarım olanakları ile yenilikçi bir yaklaşım sağlar. Bu nedenle, finisaj işlem basamağında bir çapraz bağlayıcı ve UV absorban eşliğinde kullanılan termokromik pigmentlerin (soğuk ve dokunma aktiviteli) yaşlanma davranışları incelenmiştir. Termokromik derilerin renk değiştirme özellikleri sadece sıcaklık ve sıcaklık ile bağıl nemin beraber etkisinin incelendiği hızlandırılmış yaşlanma testlerinin öncesi ve sonrasında spektrofotometrik olarak belirlenmiştir. Sonuç olarak, termokromik deriler üzerine yaşlanmanın anlamlı bir etkisi tespit edilememiştir. Fakat soğuk aktiviteli derilerde kısmi bir azalma belirlenmiş ve bu solma UV absorban ve çapraz bağlayıcı kullanımı ile engellenmiştir.

Anahtar Kelimeler: Hızlandırılmış yaşlanma, termokromik deriler, renk değiştirme etkisi, renk ölçümü

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1. INTRODUCTION

Thermochromic dyes and pigments are of vital importance for functional and aesthetic design of smart textile and leather materials and they consist of three main components: a color former (the leuco dye), a color developer (acid) and a low melting point solvent [1-4]. They are often identified as pigments instead of dyes and mostly found in encapsulated form [1-5], they change color in special activation temperatures of the colorant and the change occurred in color is reversible [1-7]. The important common applications of thermochromic pigments are in the area of sun-screening ophthalmics, security printing, optical data storage, food packaging, medical and textile engineering including protective and sports clothing [1-6]. However, standard activation temperature ranges of thermochromic pigments are lead to a limitation in the potential application areas of the pigments [1-7].

Thermochromic color changing technology offers designers to create new, innovative and smart materials by the use of different kind of fabrics including leather [4, 5], however the drawbacks of thermochromic dyes have pretended to have a tendency to restrict their use in many different use of areas. The most common problems associated with thermochromic dyes and pigments are known as color fading and poor fastness properties such as light, wash and rub fastness due to the low affinity of thermochromic pigment microcapsules [8, 9]. The fading of any kind of pigments and dyes are affected by external parameters such as oxygen, moisture, additives, surface properties [10] and the main responsible environmental factors of the fading and ageing phenomena are known as temperature and humidity. Regarding the thermochromic leathers, ageing effect could be associated with the color fading. Besides, the change occurred in these factors are lead to effect the aesthetic and performance properties as well as product life and usage areas of leather materials [11]. Nevertheless, the undesirable effect of ageing on thermochromic leathers could be prevented by the use of different chemicals that enhanced the fastness properties of thermochromic leathers [2,12,13].

Although several applications of thermochromic dyes and pigments have been found in textile industry [5-7; 14,15], limited number of studies has been published about thermochromic applications in the area of leather engineering [1,2,4,16]. Up to our knowledge except from the studies regarding the application of cold and touch activated thermochromic pigments in leather finishing process [1,2,4], Tamilmani et al., 2015 has reported a study about the synthesis and application of a metal complex dye which has a color changing temperature between 200-210^{o}C in leather finishing process for the use of safety leather products [16]. According to our knowledge this is the first study describing the ageing behaviors of thermochromic leathers under different accelerated ageing conditions including heat and humidity.

In this study, two thermochromic pigments, providing color change at 15°C (cold activated) and 31°C (touch activated) were applied in the base coat of the finishing process along with a commercial UV absorbent and a crosslinker. The color measurements of the thermochromic leathers were determined by spectrophotometrically prior and subsequent to the accelerated ageing tests performed under two different conditions (only heat and the combination of heat and moisture). The product life and the quality performance of the thermochromic leathers regarding the color changes has been questioned and being a subject of interest primarily in this study.

2. MATERIALS AND METHODS

2.1. Materials

Metis type crust leathers were used for the application of two thermochromic pigment dispersions (TMC Hallcrest, England) providing color change at 15°C (Blue; cold activated) and 31°C (Magenta; touch activated). The thermochromic pigments were applied at the base coat of the finishing process by the addition of a commercial UV absorbent (a mixture of benzotriazole and benzophenone) and crosslinker (aziridine type commercial product) in the finishing process. For the finishing application, chemicals with high UV resistance were selected as well as the solvent free waterborne acrylic resin used as a binder. Microencapsulated thermochromic pigments are in the form of water-based dispersion with the following features: solid content 48±2%, granule size 97% <6um, pH 5.0-5.5, 1-2 light fastness [1,2,4].

2.2. Methods

2.2.1. Application of thermochromic pigments

Prior to finishing applications, crust leathers were dyed with a suitable base colors (yellow for magenta thermochromic pigment and red for blue thermochromic pigment) to ensure the transition from one color to another. After drying and mechanical processing, thermochromic pigments were applied to leathers at the base coat of the finishing process [1,2,4].

Thermochromic and conventional pigment proportions were selected as 2:1 for the finishing applications and 40 units of UV absorbent was used along with the pigment application at the base coat. Besides, four different proportions (15, 20, 50 and 70 units) were used with and without cross-linker application at the top coat of the finishing procedure to improve the fastness properties of thermochromic leathers (Table 1) [4].

After thermochromic finishing applications, sampling and conditioning of the thermochromic leathers were performed in accordance with the standards of ISO 2418 and ISO 2419 respectively [17,18].

Table 1. Experimental design of thermochromic finishing applications

Trials	Base Coat	Top Coat
1	50 conventional pigment	Only top coat
2	50 conventional pigment + Clink	Top coat + Clink
3	50 pig + termchr.(1/2)	Only top coat
4	Trial 3+ Clink	Top coat + Clink
5	Trial 3 + Abs	Only top coat
6	Trial 3 + Abs + Clink	Top coat + Clink
15	Trial 3 + Abs	Top coat + 15 UV Abs
15.C	Trial 3 + Abs+ Clink	Top coat + 15 UV Abs + Clink
20	Trial 3 + Abs	Top coat + 20 UV Abs
20.C	Trial 3 + Abs + Clink	Top coat + 20 UV Abs + Clink
50	Trial 3 + Abs	Top coat + 50 UV Abs
50.C	Trial 3 + Abs + Clink	Top coat + 50 UV Abs + Clink
70	Trial 3 + Abs	Top coat + 70 UV Abs
70.C	Trial 3 + Abs + Clink	Top coat + 70 UV Abs + Clink

*Clink; Crosslinker; Abs; Absorbent

2.2.2. Accelerated ageing test

The accelerated ageing test was performed under two different conditions (only heat; 24h at 60^{o}C and the combination of heat and moisture; 24h at 50^{o}C and 90% humidity) in accordance with TS EN ISO 17228 [19].

2.2.3. Color measurements of thermochromic leathers

The color change effect acquired on the leathers was determined by spectrophotometric measurements performed by Minolta CM-3600A (Konica, Japan) prior and subsequent to accelerated ageing tests performed under different conditions of heat and humidity. Measurements were carried out according to the CIE Lab color system [20] and multiple surface color measurements (minimum 10 measurements) from different regions of the leathers were performed. These measurements were carried out separately for cold and touch activated leathers in duplicates and they were given as mean value ± standard deviation.

3. RESULTS AND DISCUSSION

The color change of thermochromic leathers aged artificially under two different conditions such as 24h at 60^{o}C and 24h at 50^{o}C and 90% humidity were evaluated according to CIE Lab color system and the L*a*b coordinates are explained as L* = 0 yields black and L* = 100 yields white; negative values of a* indicate green, positive values indicate red; negative b* values indicate blue and positive values indicate yellow [21]. The display of thermochromic leathers produced with cold and touch activated thermochromic pigments used in the leather finishing process with the combination of conventional pigments, UV absorber and cross-linker are shown in Figure 1.

The effect of ageing conditions on color values (L*a*b coordinates) of cold and touch activated thermochromic leathers are presented in Table 2-5 in inactive and active form of the leathers. In the figures, only the ΔE values of the samples treated with different applications and ageing conditions were compared to reveal the color differences of thermochromic leathers occurred after the ageing. And the difference of the ΔE value is defined as the indicator of the color difference [2].

Figure 1. The display of cold activated (above) and touch activated thermochromic leathers (below)

The change in L, "a" and "b" values under different accelerated ageing conditions were found to be nearly similar in the inactive state of the thermochromic pigments for the touch activated leathers. The color values of the samples did not prominently differ from the control groups (Table 2). The ΔE values, shown in Figure 2, presented that there was not a color difference in terms of fading occurred after two different ageing performed under the conditions of 60^{o}C and 50^{o}C at 90% relative humidity but a tendency was seemed in the direction of darker colors.

Table 2. The color values of touch activated leathers in the inactive state under different ageing conditions

	L	a	b	L	a	b	L	a	b
	Before ageing			**After the ageing at 60°C**			**After the ageing at 50°C, 90% RH**		
1	73.27±0.41	-2.93±0.48	66.59±1.08	73.35±0.33	-2.57±0.15	66.78±0.21	73.77±0.23	-2.23±0.05	67.49±0.40
2	73.27±0.54	-2.56±0.35	66.41±1.10	73.52±0.40	-2.42±0.06	67.31±0.43	73.28±0.76	-1.88±0.46	66.59±1.24
3	72.33±1.71	-3.01±0.92	59.05±1.84	71.27±1.21	-3.09±0.40	58.38±1.82	71.72±0.74	-3.12±0.20	58.15±1.63
4	73.09±0.10	-2.58±0.55	59.89±0.25	69.81±0.87	-2.18±0.62	56.56±0.78	70.38±1.35	-2.24±0.63	57.81±1.76
5	71.11±0.84	-3.29±0.67	56.90±2.60	71.73±0.66	-3.67±0.16	57.59±1.11	72.19±0.88	-3.72±0.18	58.47±0.34
6	71.24±1.15	-3.28±0.09	56.88±1.46	71.46±0.65	-3.20±0.30	57.90±1.06	71.68±0.72	-3.47±0.05	57.82±0.60
15	72.5±0.58	-3.75±0.30	56.92±1.45	71.58±0.66	-2.84±0.66	57.96±0.98	71.35±0.78	-2.47±0.26	57.50±1.02
15.C	71.37±0.32	-3.32±0.91	56.28±1.48	70.78±0.62	-3.25±0.73	55.95±0.48	70.99±0.67	-3.45±0.64	55.95±0.79
20	72.19±0.38	-3.39±0.36	57.13±0.83	71.89±0.24	-2.77±0.61	57.91±0.62	71.84±0.71	-3.62±0.27	57.27±1.09
20.C	70.92±0.93	-3.13±0.42	55.77±1.07	71.51±0.32	-3.25±0.30	56.81±0.45	71.71±0.13	-3.09±0.30	57.07±0.51
50	71.79±0.42	-2.97±0.83	57.15±1.67	71.66±0.33	-2.85±0.52	56.68±1.76	71.19±0.90	-2.87±0.74	56.86±1.87
50.C	71.13±0.69	-3.26±0.71	55.97±2.27	71.87±0.44	-3.93±0.62	57.32±2.14	71.77±0.42	-3.84±0.56	57.03±2.37
70	71.46±0.46	-3.50±0.64	56.88±1.19	71.63±0.30	-2.93±1.46	58.02±0.94	71.56±0.42	-3.13±1.29	57.72±1.44
70.C	71.52±0.32	-3.66±0.39	56.61±2.35	71.40±0.90	-3.69±0.77	56.74±2.29	70.90±0.89	-3.63±0.58	56.67±2.53

n=10

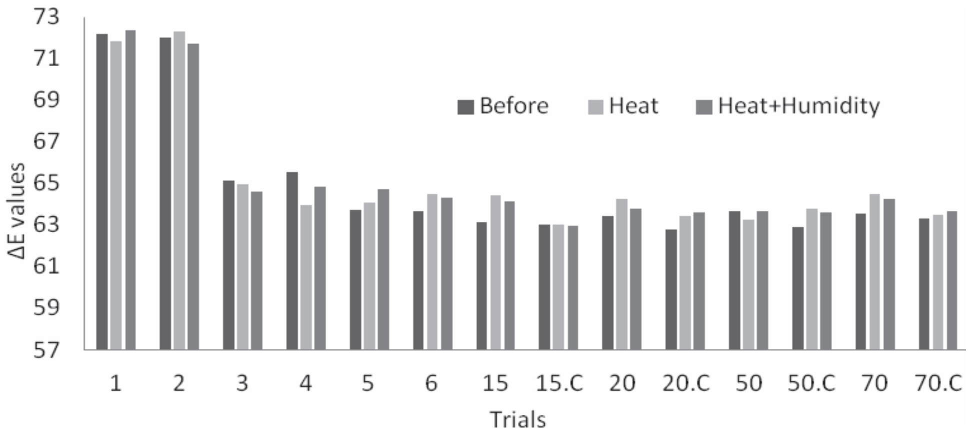

Figure 2. ΔE values of touch activated leathers under different ageing conditions in their inactive state

Table 3. The color values of touch activated leathers in the active state under different ageing conditions

	L	a	b	L	a	b	L	a	b
	Before ageing			**After the ageing at 60°C**			**After the ageing at 50°C, 90% RH**		
1	73.27±0.41	-2.93±0.48	66.59±1.08	73.35±0.33	-2.57±0.15	66.78±0.21	73.77±0.23	-2.23±0.05	67.49±0.40
2	73.27±0.54	-2.56±0.35	66.41±1.10	73.52±0.40	-2.42±0.06	67.31±0.43	73.28±0.76	-1.88±0.46	66.59±1.24
3	55.44±0.26	24.26±1.44	34.90±0.46	55.36±1.32	23.02±2.44	35.20±2.07	61.73±1.46	14.73±3.06	43.34±1.79
4	55.54±0.55	24.57±0.66	34.71±0.40	55.04±0.56	22.17±0.73	34.85±1.08	61.62±2.26	13.51±4.55	44.46±3.46
5	58.29±0.48	20.31±0.71	38.38±0.84	58.54±1.10	19.21±1.27	38.31±0.56	62.38±0.90	14.19±2.49	43.96±2.25
6	58.08±1.43	19.98±0.86	37.42±0.76	58.21±1.60	19.41±2.38	38.47±1.40	62.82±1.81	12.75±2.55	44.82±1.48
15	59.79±0.8	17.87±1.70	39.34±0.38	57.90±1.35	20.60±0.71	37.87±1.84	61.96±1.59	14.58±1.11	43.51±2.15
15.C	58.04±0.78	19.58±1.77	36.85±0.34	58.36±0.25	18.10±0.60	37.93±0.60	62.85±0.93	11.61±1.31	44.02±2.26
20	59.06±0.62	18.98±1.01	38.36±0.40	58.29±1.25	20.36±1.27	38.14±1.27	61.43±1.61	15.46±2.00	42.08±1.95
20.C	58.62±0.58	18.28±0.78	38.07±0.46	65.61±7.08	7.20±12.35	48.14±9.89	63.87±1.89	11.44±3.39	45.43±2.73
50	58.80±1.12	19.05±1.80	38.39±0.28	59.60±0.41	18.29±1.11	39.09±1.34	61.76±0.56	14.36±1.82	42.93±0.97
50.C	58.23±1.02	18.46±2.37	38.00±0.38	59.01±0.35	18.65±1.29	38.64±1.42	62.40±0.68	12.99±2.33	43.56±0.75
70	58.45±1.26	18.60±2.40	38.53±1.02	58.49±1.12	19.74±0.97	38.97±1.79	60.66±0.75	16.26±0.62	41.87±1.70
70.C	58.04±2.21	18.69±3.65	37.96±1.20	59.10±2.25	17.81±2.86	38.82±1.10	60.30±2.02	15.54±3.13	41.38±1.64

n=10

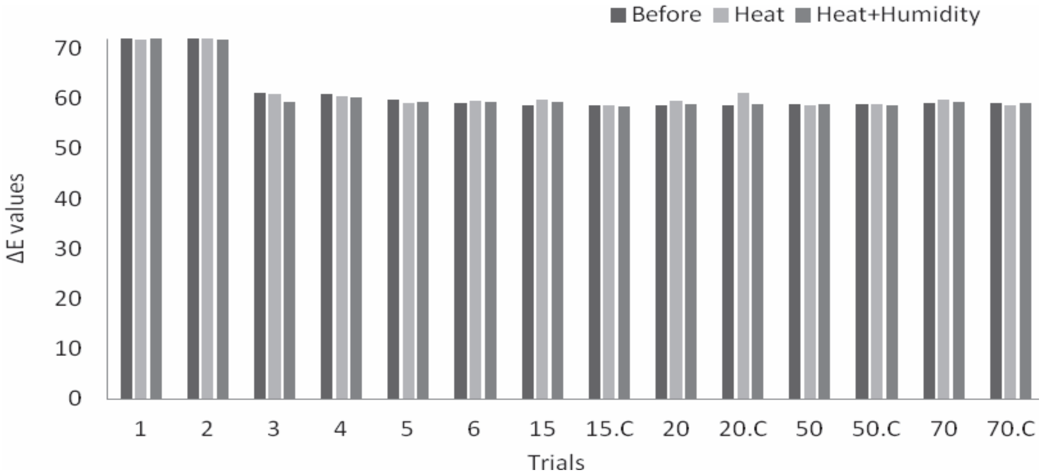

Figure 3. ΔE values of touch activated leathers under different ageing conditions in their active state

The color measurement values of touch activated leathers in their active state in terms of L, a, b and ΔE values are given in Table 3 and Figure 3 respectively.

The L values, which are associated with the brightness, were not found different from the control groups measured prior to ageing process for the heat ageing. But for the heat and humidity ageing the brightness values were found higher than the heat ageing results as well as control groups. The positive a values are the indication of red color that show the thermochromic effect of touch activated thermochromic pigments. No remarkable difference was observed after the heat ageing, although heat and humidity ageing caused a decrease in the values of a. When the b values of the touch activated leathers in the active state were evaluated, an opposite situation was determined for the heat and humidity ageing compared to redness values (Table 3).

The ΔE values of touch activated leathers, corresponds to color difference, also indicated that the thermochromic characteristics of touch activated leathers in the active state were maintained after the accelerated ageing processes performed under heat and humidity and this color changing effect was still reversible (Figure 3).

Different ageing conditions did not influenced the L, a, b coordinates apparently for the cold activated thermochromic leathers in their inactive state (Table 4). Besides, the ΔE values of the cold activated leathers gave similar results to touch activated leathers in the inactive state. A color difference was observed according to Figure 3 but this was not seemed in a direction of fading, it was more likely to lead darker colors (Figure 4).

Table 4. The color values of cold activated leathers in the inactive state under different ageing conditions

	L	a	b	L	a	b	L	a	b
	Before ageing			**After the ageing at 60°C**			**After the ageing at 50°C, 90% RH**		
1	35.72±0.71	39.58±1.90	14.13±0.54	35.92±0.19	39.72±0.46	14.57±0.61	36.19±0.20	40.01±0.36	14.49±0.70
2	35.85±0.41	38.72±0.71	13.58±0.69	35.86±0.26	38.69±0.46	13.98±0.25	36.16±0.17	39.44±0.34	14.08±0.10
3	38.42±0.42	40.25±0.40	11.07±0.86	37.98±0.67	40.17±0.85	11.89±0.15	38.20±0.48	39.68±0.61	11.57±0.27
4	38.20±0.56	39.49±0.79	10.88±0.72	38.02±0.27	39.42±0.47	11.46±0.41	37.78±0.55	38.89±1.00	11.28±0.22
5	38.97±0.65	38.51±0.63	9.31±1.44	39.16±1.55	38.39±0.14	9.82±1.06	38.03±0.85	37.26±0.63	9.71±0.44
6	38.99±0.41	38.49±1.78	9.37±1.49	38.39±0.14	38.18±1.40	10.27±0.87	37.91±0.72	37.17±0.80	9.52±0.43
15	38.48±1.05	38.24±0.64	9.46±1.26	38.76±0.38	38.78±0.46	10.15±0.44	37.83±0.51	37.37±0.91	9.82±0.46
15.C	38.28±0.52	36.82±1.29	8.97±1.13	38.30±0.36	37.78±0.46	9.91±0.14	38.35±0.62	37.55±0.38	9.38±0.34
20	39.20±0.92	38.21±0.97	8.49±0.43	39.61±0.61	39.23±0.77	9.91±0.55	39.40±0.57	38.21±0.34	9.27±0.36
20.C	38.73±0.58	37.84±1.11	9.02±1.28	38.92±0.43	38.47±0.14	9.39±0.43	38.41±0.46	37.75±0.60	9.20±0.25
50	37.76±1.08	36.70±1.01	8.92±0.53	39.59±2.01	36.76±0.48	8.95±0.62	39.24±0.67	37.04±0.67	9.41±0.40
50.C	38.52±0.29	37.90±0.66	9.23±0.79	38.13±0.65	38.09±1.20	10.23±0.39	37.93±0.57	37.82±0.78	10.06±0.34
70	38.2±0.36	38.00±1.08	9.57±1.42	38.51±0.96	38.95±0.22	10.51±0.80	38.07±1.06	37.81±1.77	10.23±0.14
70.C	37.86±0.50	37.14±1.26	9.65±0.98	38.60±0.18	38.60±1.17	10.33±0.57	37.62±0.45	37.23±1.28	10.06±0.80

n=10

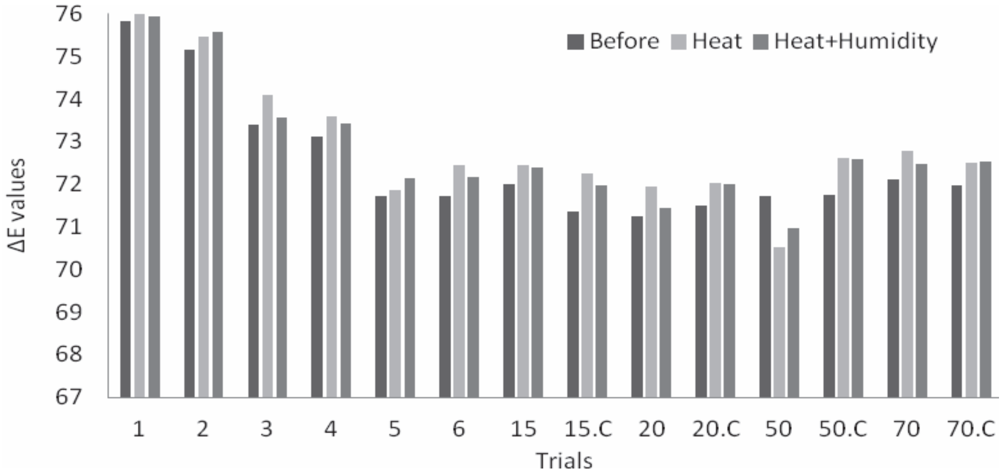

Figure 4. ΔE values of cold activated leathers under different ageing conditions in their inactive state

Table 5. The color values of cold activated leathers in the active state under different ageing conditions

	L	a	b	L	a	b	L	a	b
	Before ageing			**After the ageing at 60°C**			**After the ageing at 50°C, 90% RH**		
1	35.72±0.71	39.58±1.90	14.13±0.54	35.92±0.19	39.72±0.46	14.57±0.61	36.19±0.20	40.01±0.36	14.49±0.70
2	35.85±0.41	38.72±0.71	13.58±0.69	35.86±0.26	38.69±0.46	13.98±0.25	36.16±0.17	39.44±0.34	14.08±0.10
3	29.77±0.65	19.89±2.22	-1.22±0.29	29.17±1.01	19.52±2.09	-1.34±0.43	30.51±0.26	21.29±0.48	-0.07±0.26
4	30.14±0.99	19.96±1.44	-0.80±0.86	29.73±0.81	18.49±3.34	-1.26±0.32	30.33±0.37	20.10±1.01	-0.03±0.19
5	30.59±0.95	20.54±1.74	-1.92±0.60	31.28±2.35	21.06±2.63	-1.83±0.72	30.69±0.88	20.62±0.37	-1.28±0.62
6	30.85±0.43	21.06±0.71	-1.69±0.13	29.95±0.61	19.47±1.24	-2.15±0.39	29.78±0.61	19.30±1.38	-1.78±0.33
15	29.25±0.54	20.78±3.57	-2.35±0.57	30.47±0.42	20.25±0.74	-2.03±1.09	30.43±0.16	20.03±0.91	-1.42±0.54
15.C	30.50±0.52	19.75±1.90	-1.71±0.45	30.16±0.37	18.96±0.70	-2.08±0.37	30.80±0.84	20.54±1.64	-1.70±0.53
20	31.12±0.37	22.14±0.93	-2.89±0.34	32.44±1.92	25.20±4.05	-0.30±2.79	33.48±2.05	26.51±4.43	0.48±2.94
20.C	31.13±0.60	20.85±2.05	-1.78±0.38	30.38±0.51	21.09±2.00	-2.87±0.28	30.54±0.31	20.80±1.24	-2.40±0.24
50	30.23±0.97	20.79±1.39	-1.93±0.35	32.36±3.28	23.87±3.49	-0.25±2.01	33.03±2.27	25.30±4.14	1.07±3.20
50.C	30.56±0.45	20.65±1.25	-2.08±0.29	29.18±0.69	18.40±1.40	-1.58±0.29	29.41±0.75	19.67±1.53	-1.89±0.10
70	30.36±1.11	20.34±3.04	-2.43±0.26	29.52±1.53	19.04±2.95	-2.36±0.23	29.94±0.97	19.12±2.80	-1.64±0.35
70.C	30.12±0.58	19.06±0.86	-1.85±0.57	29.09±1.19	18.18±1.96	-2.24±0.36	29.74±1.05	18.61±0.25	-1.62±0.30

n=10

The color values of cold activated leathers in the active state are shown in Table 5. There was no remarkable difference of accelerated ageing conditions on thermochromic leathers for the brightness and redness values. But for the b value, which represents the blue color, was affected from the ageing conditions in a different way. A decrease was observed for the b values that was representing the color fading. But the increase of UV absorbent at the top coat of the finishing process along with the cross-linker use prevented the color fading occurred under the conditions of heat ageing. However, the cold activated leathers in their active state were influenced intensively from the heat and humidity ageing conditions compared to heat ageing (Table 5).

The ΔE values of cold activated thermochromic leathers are given in Figure 5 and these results were found not similarly with the results obtained from the touch activated leathers in the active state. The ΔE values of cold activated leathers were intensively affected from the ageing conditions compared to touch activated leathers. The use of cross-linker and the higher amounts of UV absorbent prevented the color fading and color differences of the thermochromic leathers. Especially the use of UV absorbent in the proportion of ≥20 could be the solution to avoid the low fastness properties of cold activated thermochromic leathers.

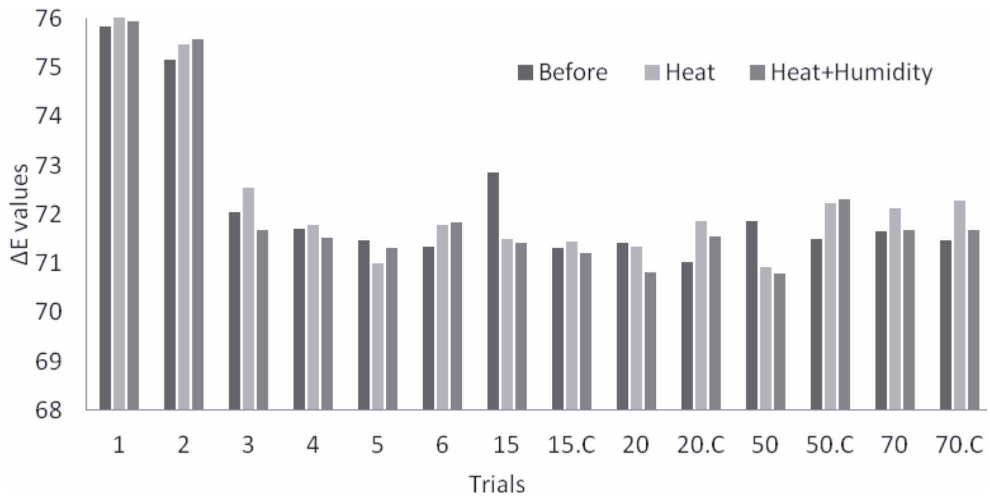

Figure 5. ΔE values of cold activated leathers under different ageing conditions in their active state

4. CONCLUSION

In this study, the accelerated ageing behaviors of touch and cold activated thermochromic leathers were aimed to investigate by two different accelerated ageing conditions such as only heat and heat associated with relative humidity to ensure the quality performance of the new fashion thermochromic leathers in terms of color fading and the following conclusions have been drawn;

a) No negative effect of heat and heat and humidity accelerated ageing conditions was found on touch activated thermochromic leathers and the thermochromic effect was still maintained although a little decrease for redness and a little increase for yellowness values were determined after heat and humidity ageing in the active state of touch activated leathers.

b) Similar color measurement results were obtained from cold activated leathers in the inactive states of the pigments for the two ageing conditions and the thermochromic effect of the pigments was protected.

c) However, lower color measurement values, especially for b values, were determined from the cold activated leathers in their active state and with the addition of an UV absorbent and cross-linker, the color fading was prevented for both ageing conditions, although ageing conditions of heat and humidity affected the color fastness properties of leathers intensively.

d) When the thermochromic leathers were evaluated organoleptically after ageing, it could be revealed that color changing effect of thermochromic leathers was existed for touch and cold thermochromic leathers.

e) Consequently, it was revealed that thermochromic pigments could be alternatively used in leather finishing applications according to leather fashion trends in addition to technical leathers and textiles in the field of medical and protective wear applications.

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