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Preparation of Temperature-Sensitive Colour-Changing Materials and Their Applications to Clothing Fabrics: Taking a Compound Composed of Crystal Violet Lactone, Bisphenol A, and Octadecanol as an Example

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ABSTRACT

A compound was prepared using crystal violet lactone, bisphenol A, and octadecanol. An in-situ polymerization method was used to prepare temperature-sensitive colour-changing microcapsules. It was printed on fabrics using printing technology. The colour-changing performance of the composite material, thermal weight changes of the microcapsules, and properties of the fabric coated with microcapsules were analyzed using cases. Printed fabric properties of colour fastness to washing, rubbing, strength and colour strength (K/S) were measured in terms of binder (%), a crosslinking agent (%), and temperature variations. The results indicated that the compound gradually changed from purple to blue within the temperature range of 3 °C to 60 °C and eventually faded into a colourless state. The weight of the microcapsule powder decreased as the temperature rose, with no significant change in weight before reaching 100 °C. Between 100 °C and 200 °C, there was a rapid decrease in weight, followed by a slow decrease between 200 °C and 350 °C. After reaching 350 °C, the weight tended to stabilize. When the mass fraction of the cross-linker was 3%, the mass fraction of the binder was 25%, and the baking temperature was 120 °C, the colour fastness, fabric strength, and K/S value (colour intensity) of the cotton fabric coated with microcapsules were optimal.

KEYWORDS

temperature-sensitive colour-changing materials, clothing fabrics, microcapsule, printing

INTRODUCTION

The progress of science and technology has opened up new avenues for clothing design, with temperature-sensitive colour-changing materials being one of the prominent technologies [1]. The main principle behind these materials is their ability to undergo molecular structural changes in response to temperature fluctuations, leading to alterations in the material's reflectance spectrum and resulting in surface colour changes [2]. Compared to conventional materials, temperature-sensitive colour-changing materials exhibit rapid and dynamic colour transformations, presenting various colour options in apparel design at different temperatures [3]. When the temperature returns to its initial state, the colour of the material also reverts to its original state. The use of temperature-sensitive

colour-changing materials in clothing fabrics caters to the individual preferences of consumers. Moreover, using different colours at varying temperatures can add interest and enhance the attractiveness of clothing design. For instance, Sahebkar et al. prepared thermochromic fibres by incorporating single strands of poly(methyl methacrylate) into thermochromic powder using electrostatic spinning [4]. Hakami et al. investigated the influence of surfactants on the TiO_2 microencapsulation of thermochromic materials [5]. Their experimental results verified the reversible thermochromic behaviour. A new approach was introduced by Bao et al. to produce cotton fabrics through the utilization of mercapto click chemistry [6]. Their findings demonstrated the successful synthesis of photochromic materials, and the resulting cotton fabrics exhibited significant and rapid colour changes under ultraviolet radiation and heating, showcasing excellent photochromic properties. This paper provides a brief overview of thermochromic materials. Subsequently, a thermochromic compound was prepared using crystal violet lactone, bisphenol A, and octadecanol. The in-situ polymerization was employed to create thermochromic microcapsules, which were then applied to clothing fabrics using printing technology. The fabrics underwent performance tests to assess their properties.

EXPERIMENTAL ANALYSIS

Experimental Materials and Apparatus

Materials: crystal violet lactone (a colourant that provides colours), bisphenol A (a colour-developing agent that determines the shade of microcapsule colour), octadecanol solvent (a solvent determining the colour-changing temperature), formaldehyde (used for preparing microcapsule wall materials), melamine (used for preparing microcapsule wall materials), triethanolamine (a pH adjuster for preparing wall materials), citric acid (a pH adjuster), binder (used for binding microcapsules with cloth), cross-linker (used for strengthening the binding between microcapsules and cloth, and cotton cloth).
Instruments: electronic balance, constant-temperature magnetic stirrer, constant-temperature water bath, electric blast drying oven, and translational room temperature small sample machine [7].

Preparation Methods

The basic process of preparing a temperature-sensitive colour-changing material as well as a fabric is shown in Figure 1.

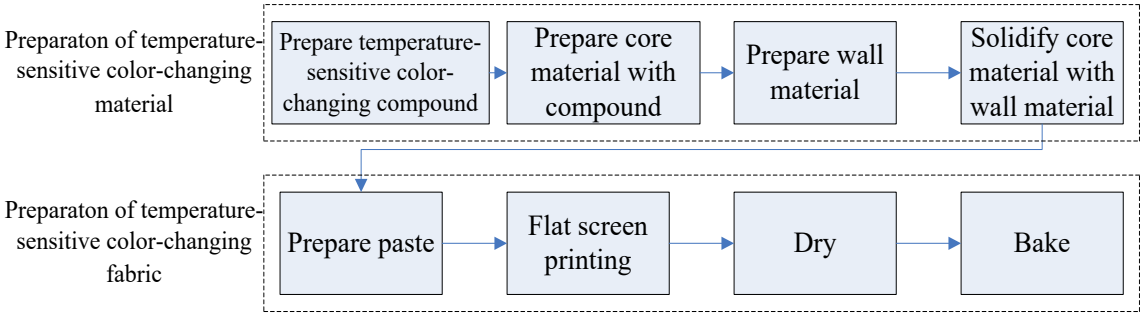

Figure 1. The preparation process of the temperature-sensitive colour-changing material and fabric

The specific steps are described below.

1. According to the mass ratio of 1:3:40, crystal violet lactone, bisphenol A, and octadecanol were weighed and placed into a beaker. Then, a water bath with a constant temperature of 70 °C was used for heating. When the octadecanol in the beaker was completely melted, the temperature-sensing colour-changing compound was obtained after one hour using a constant temperature magnetic stirrer [8].
2. The core material was prepared using the compound. Deionized water, emulsifier, and compound were weighed in a mass ratio of 30:2:1 and placed into a beaker. They were mixed using a high-speed shear mixer in a constant temperature water bath set at 70 °C, with a mixing speed of 6,500 rad/min for 250 min.
3. To prepare the wall material, deionized water, a 37% formaldehyde solution, and melamine were mixed in a mass ratio of 20:5:2. The resulting mixture was then transferred into a beaker, and its pH level was adjusted to 8.5 using triethanolamine. The solution was then stirred in a constant temperature water bath set at 70 °C until it became clear [9].
4. The core material was solidified using the wall material. The wall material solution was gradually introduced into the emulsion of the core material using a burette, with continuous stirring throughout the process. Once the entire wall material solution had been added to the core material emulsion, the pH of the mixture was adjusted to 6.5. Subsequently, it was transferred to a round-bottom flask and subjected to pH adjustments every 60 minutes in a constant-temperature water bath maintained at 70 °C. This procedure was repeated three times in total. During the adjustment, the mixture was constantly stirred. When the pH reached 3.5 and had been reacting for 60 minutes, the reaction was stopped. Then, the mixture was filtered and dried in a 60 °C oven to obtain thermochromic microcapsules [10].

The steps above outline the process of preparing the thermochromic material. Then, it was applied to clothing fabrics using printing [11]. The specific steps are shown below.

5. The paste was prepared by mixing the microcapsules, binder, cross-linking agent, and distilled water in a specific ratio (refer to the experimental project later for the specific ratio). The binder is composed of water-based polyurethane glue, while the cross-linker consists of polyacrylic ester.
6. Flat screen printing was performed by evenly printing the paste onto the clothing fabric using a translational room-temperature hand sample machine [12].
7. The fabrics were pre-dried using an oven at a temperature of 80 °C for 2 min.
8. The fabrics were baked using an oven.

Preparation Parameters

The temperature-sensitive colour-changing microcapsules, which were self-made, were used to produce a printing paste. This paste was then printed onto the fabric of clothing to achieve a temperature-sensitive colour-changing effect. In this experiment, various quantities of binder and crosslinking agent were used, along with varying baking temperatures, to examine their impact on the fabric's performance. The preparation scheme was designed based on three aspects: the amount of binder, the amount of crosslinking agent, and the baking temperature.

Table 1. Three test scenarios

	Test Scenario 1	Test Scenario 2	Test Scenario 3
Microencapsulated mass/g	1	1	1
Crosslinking agent mass fraction/%	3	1, 2, 3, 4, 5	3
Binder mass fraction/%	10, 15, 20, 25, 30	25	25
Distilled water/g	x	x	x
Total mass of paste/g	20	20	20
Baking temperature/°C	120	120	110, 120, 130, 140, 150
Baking time/min	4	4	4

Performance Test

Temperature-Sensitive Colour-Changing Microcapsule Test

(1) Test of apparent form and colour-changing performance

Scanning electron microscopy was used to observe the surface morphology of thermochromic colour-changing microcapsules. The colour-changing performance mainly depends on the compound in the core material. Therefore, for ease of observation, direct testing was conducted on the solution of the compound in the core material. Firstly, the compound solution was absorbed by filter paper at 70 °C. After the filter paper cooled down, it was placed on a heating device and heated. During this process, the colour of the filter paper was captured using a camera, and its surface temperature was

simultaneously measured with an infrared thermometer. Colours were expressed by the CIE-LAB values of colours obtained using the colour picker function in Photoshop software.

(2) Test of heat resistance

An appropriate amount of homemade thermochromic microcapsule powder was taken and placed into a thermogravimetric analyzer (HTG-1). The heating rate was set to 10 °C/min, and the sample was heated in a nitrogen environment. Once the instrument was activated, the relationship between sample weight and temperature was recorded.

Temperature-Sensitive Colour-Changing Fabric Performance Test

Colour fastness test, fabric strength test, and fabric discolouration performance test were carried out on the prepared fabric. The colour fastness test follows the standards GB/T3920-2008 and GB/T3921-2008 [13] to assess the colour fastness of the fabric. This is done using a dyeing friction colour fastness tester and a washing fastness tester. During the testing process, the fabric was subjected to dry rubbing, wet rubbing, and washing methods. In the dry rubbing method, the fabric was cut into small pieces measuring 12 cm × 6 cm and placed on a friction colour fastness tester. After applying a specific pressure, it was rubbed against a standard friction white cloth. Then, a grey chip was used to rate the colour transfer onto the standard friction white cloth. The steps of the wet rubbing method were similar, but the difference lay in both pieces of fabric used for friction being moistened with water. The water-washing method involves sewing the cut fabric together with a standard lining fabric and then placing it into a colourfastness tester for washing, rinsing, and drying. During this process, an appropriate amount of stainless steel beads were added to expedite progress. Afterwards, a grey chip was used to grade the colours transferred onto the standard lining fabric.

The fabric strength test follows the GB/T19976-2005 standard to measure the bursting strength of the fabric using a fabric strength machine [14]. During the testing process, the fabric was first cut into small pieces. Then, a short incision was made in the centre of each piece. Subsequently, each sample was placed in the testing apparatus, with its incision positioned beneath a circular test head. An increasing vertical downward force was applied until the fabric tore apart. Throughout this procedure, both the maximum force required to tear the fabric and the displacement at which tearing occurred were recorded.

Before testing the colour change performance of the fabric, the fabric was first cut into rectangles with specifications of 12 cm × 6 cm. Then, the *K/S* value of the fabric at room temperature was determined using a colour measuring and matching meter. Then, the fabric was heated up to 55 °C using a thermostatic metal bath and maintained at that temperature for 90 s, followed by the detection of the *K/S* value [15]. *K/S* the value represents the ratio of the absorption coefficient (K) of the colouring

material in a dyed cloth to the scattering coefficient (S), which indicates the shade of colour on the surface of a solid sample. A higher value indicates better dyeing performance of the dye [16].

Test Results

Test Results of Microcapsule Morphology and Colour-changing Performance

The results of the temperature-sensitive colour-changing microcapsules observed under a scanning electron microscope (SEM) are shown in Figure 2. It can be observed that, despite slight variations in size, the prepared microcapsules exhibited a spherical and evenly distributed overall shape, with an average particle size of 28.45 μm. Figure 3 illustrates the colour change of the compound within the microcapsule core, which is dependent on temperature. It is evident from Figure 2 that as the temperature increased, the colour of the complex gradually transitioned from purple to blue and subsequently faded. Table 2 shows the CIE values of the colour in the complex with temperature variations. It can be observed that as the temperature increased, the L value of the complex colour also increased, while the a value initially rose and then decreased, and the b value first decreased and then rose.

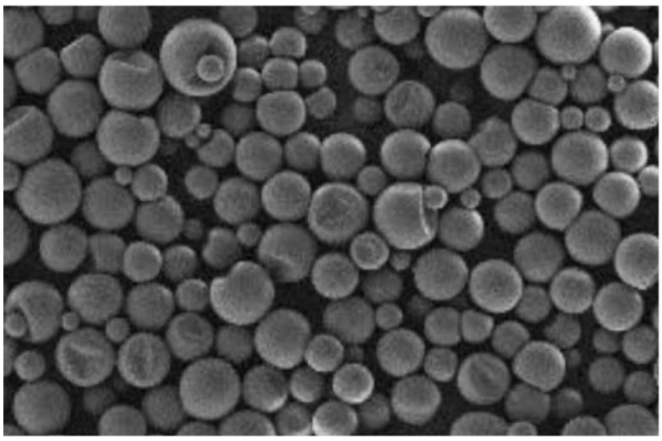

Figure 2. A SEM image of temperature-sensitive microcapsules

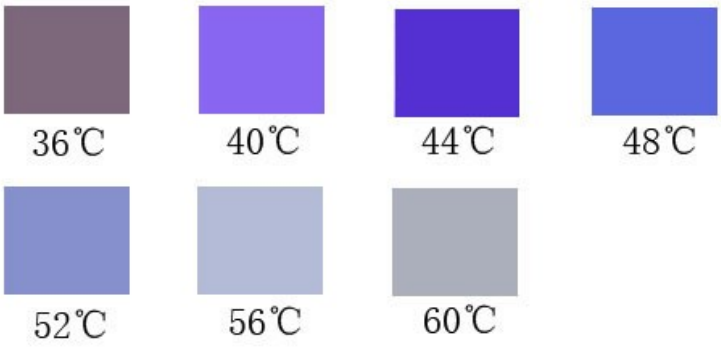

Figure 3. Colour variation of microcapsule core material compound with temperature change

Table 2 Changes in the CIE value of the colour of the core material complex with temperature variations

	Temperature, °C		36	40	44	48	52	56	60
Colour		L^*	46	52	34	48	61	76	71
		a^*	11	38	49	23	7	2	0
		b^*	-8	-66	-79	-64	-33	-15	-7

The Heat Resistance of Microcapsules

The weight change curve of microencapsulated powder in a gradually increasing temperature environment is shown in Figure 4. It can be observed that as the environmental temperature increased, the weight of the microencapsule powder gradually decreased. However, before reaching 100 °C, the weight reduction of the microcapsules was relatively small. This is because the temperature at this point was not high enough to cause the rupture of microcapsules; it only led to water evaporation. After reaching a temperature above 100 °C, the weight of the microcapsules decreased rapidly. This was attributed to incomplete encapsulation of the core materials by the wall materials during the preparation process. These partially encapsulated core materials undergo rupture and escape when exposed to high temperatures. After reaching 200 °C, the weight of the microcapsules decreased slowly. This was because, although the core material underwent decomposition at high temperatures, the encapsulating material partially prevented its escape. However, after reaching 350 °C, the weight reduction of microcapsules accelerated again. This was because, at this temperature, the encapsulating material also became damaged and could no longer prevent the escape of the core material.

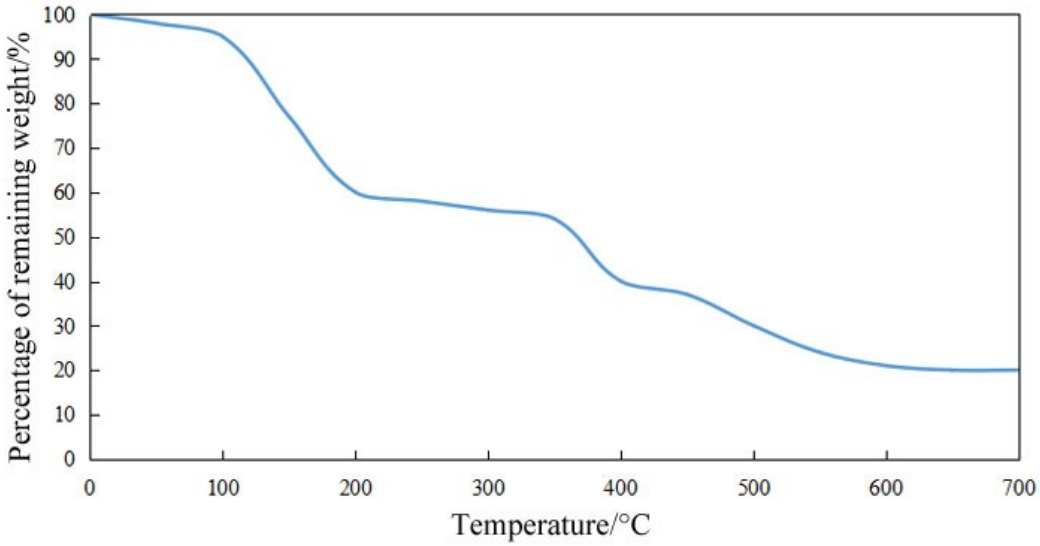

Figure 4. The thermogravimetric curve of microcapsule

Performance Test Results of the Temperature-Sensitive Colour-Changing Material

The impact of the binder mass fraction on colour fastness and fabric strength during the fabric preparation is displayed in Table 2. According to the data presented in Table 2, an upward trend can be observed in both colour fastness and fabric strength of the fabric as the mass fraction of the binder increases. However, the increase in colour fastness and fabric strength is not significant when the binder mass fraction is increased to 30%. There was no significant change in the *K/S* value of the fabric as the binder mass fraction increased under normal and high-temperature (55 °C) conditions. However, for the same binder mass fraction, the *K/S* value of the fabric at normal temperature was higher than that at high temperature.

Table 2. Effect of binder mass fraction on colour fastness, fabric strength, and *K/S* value

Binder mass fraction		10%	15%	20%	25%	30%
Colour fastness to dry rubbing/level	Warp	1	2	3	4	4
	Weft	2	1	3	3	4
Colour fastness to wet rubbing/level	Warp	1	2	3	4	4
	Weft	1	2	3	3	4
Colour fastness to washing/level	Discolour	2	3	4	4	4
	Stain	3	4	4	4	5
Fabric strength	Bursting strength/N	652	703	741	774	782
	Bursting displacement/mm	87.80	88.01	88.69	88.92	89.32
Fabric *K/S* value	Room temperature (20 °C)	3.2	3.3	3.2	3.3	3.2
	High temperature (55 °C)	0.80	0.81	0.81	0.82	0.81

From Table 3, it can be seen that the colour fastness and fabric strength of the fabric increased with an increase in the mass fraction of the cross-linker. However, the rate of change for both colour fastness and fabric strength gradually decreased after increasing to 4%. The *K/S* value of the fabric at room and high temperatures was observed to change with the increase of the cross-linker mass fraction. Specifically, the *K/S* value of the fabric at room temperature decreased, while there was no significant change at high temperature. It was also found that the *K/S* value of the fabric at room temperature was larger than the *K/S* value at high temperatures when the cross-linker mass fraction was the same.

Table 3. Effect of the cross-linker mass fraction on colour fastness, fabric strength and *K/S* value

Cross-linker mass fraction		1%	2%	3%	4%	5%
Colour fastness to dry	Warp	3	4	3	4	5
rubbing/level	Weft	3	4	3	3	4

Cross-linker mass fraction		1%	2%	3%	4%	5%
Colour fastness to wet rubbing/level	Warp	3	4	4	4	5
	Weft	2	3	3	3	4
Colour fastness to washing/level	Discolour	3	4	4	5	5
	Stain	3	4	4	5	4
Fabric strength	Bursting strength/N	653	704	742	776	781
	Bursting displacement/mm	87.81	88.11	88.72	88.98	89.31
Fabric *K/S* value	Room temperature (20 °C)	4.1	3.9	3.8	3.2	3.1
	High temperature (55 °C)	0.8	0.9	0.8	0.8	0.8

The impact of baking temperature on colour fastness and fabric strength during the preparation process is presented in Table 4. It can be seen that as the baking temperature rose, the colour fastness and fabric strength of the fabric also increased, but in general, the increase was not significant. For the *K/S* value of the fabric at room and high temperatures, there was no significant change in the *K/S* value of the fabric with the increase in baking temperature. Additionally, the *K/S* value of the fabric at room temperature was higher than that at high temperature under the same baking temperature.

Table 4. Effect of baking temperature on colour fastness, fabric strength and *K/S* value

Baking temperature		110°C	120°C	130°C	140°C	150°C
Colour fastness to dry rubbing/level	Warp	3	3	3	3	4
	Weft	2	2	3	3	4
Colour fastness to wet rubbing/level	Warp	1	1	2	3	4
	Weft	1	2	2	3	4
Colour fastness to washing/level	Discolour	3	3	3	4	4
	Stain	2	4	4	4	4
Fabric strength	Bursting strength/N	651	705	741	775	782
	Bursting displacement/mm	87.79	88.07	88.67	88.96	89.28
Fabric *K/S* value	Room temperature (20 °C)	3.2	3.3	3.2	3.3	3.2
	High temperature (55 °C)	0.80	0.81	0.81	0.82	0.81

DISCUSSION

The increasing demand for diverse apparel designs by consumers has motivated manufacturers to enhance the design of clothing fabrics. Temperature-sensitive colour-changing materials offer a unique feature in which their colour changes with temperature. This makes them an attractive choice for enhancing the variety of clothing fabrics. This paper presents the preparation of temperature-sensitive colour-changing microcapsules using the in-situ polymerization method. These microcapsules were then applied to clothing fabrics using printing technology, and the performance of the fabrics was tested. The final results are shown above. The ternary compound that makes up the core material of

microcapsules consists of crystal violet lactone, bisphenol A, and octadecanol. Crystal violet lactone serves as a colourant, providing colour for the compound. Bisphenol A acts as a colour-developing agent, altering the colour of crystal violet lactone. Octadecanol functions as a solvent for both components and determines the temperature at which the colour change takes place. The colour-changing mechanism of a compound is achieved by utilizing physical or chemical means to induce a change in the conjugation state of the compound. Octadecanol solvent is in a solid state at low temperatures. At this stage, the lactone ring in crystal violet accepts protons from bisphenol A, resulting in the formation of an open-ring structure and conjugation with bisphenol A. This interaction imparts colour to the compound. When the temperature rises, the lactone ring gradually closes, causing the conjugated state with bisphenol A to gradually disappear, resulting in a gradual colour change to transparent.

As the mass fraction of the binder increased, the colour fastness of the fabric improved. This is because the film formed during the drying process of the binder fixes the microcapsules at the fabric fibres. The greater the amount of binder, the thicker the film formed, which leads to a more secure attachment of the microcapsules to the fibres. However, an excessively thick film may affect the fabric's texture and feel. Additionally, the increase in fabric strength was also linked to the presence of the fixation film. For the change of the *K/S* value of fabrics, the amount of binder had less influence on the *K/S* value, so it was unchanged.

As the mass fraction of the cross-linking agent increased, the colour fastness of the fabric also improved. Similar to the binder, the cross-linker formed a mesh structure during the drying process. This mesh structure effectively captured a greater number of microcapsules. Moreover, the mesh structure of the crosslinking agent acted as the skeleton of the binder fixation film, enhancing the film’s strength and improving the fixation effect of microcapsules on the fabric. The mass fraction of the cross-linking agent under high temperature did not affect the *K/S* value, while under normal temperature conditions, the increase in the amount of cross-linking agent led to an increase in the *K/S* value. This is because the cross-linking agent appeared milky white under normal temperature conditions, and as its amount increased, it diluted the colour of the microcapsules.

As the baking temperature increased, the colourfastness of the fabric also increased. This is because a higher baking temperature resulted in a stronger bond between the binder and crosslinking agent, leading to improved fixation of microcapsules. However, extremely high temperatures can cause damage to the microcapsules; therefore, the baking temperature should not be too high. The *K/S* value of the fabric did not change significantly because the amount of crosslinker and binder were the same. The limitation of this study lies in the use of a controlled variable method to investigate the effects of crosslinking agent dosage, adhesive dosage, and baking temperature on fabric during the preparation of fabric coated with microcapsules. In this process, two factors were held constant; however, the

appropriateness of these fixed values was not considered. Therefore, future research will use orthogonal experimental design to assess the performance of fabrics under various combinations of factor values.

CONCLUSION

This paper briefly describes the temperature-sensitive colour-changing materials. The test introduces the preparation of the temperature-sensitive colour-changing compound using crystal violet lactone, bisphenol A, and octadecanol solvent. It is then followed by an introduction to the temperature-sensitive colour-changing microcapsules using in situ polymerization. Finally, the microcapsules were applied to clothing fabrics using printing technology, and the performance of the fabrics was tested. The results were as follows. (1) The average particle size of the microcapsules was 28.45 μm; as the temperature increased, the colour of the compound gradually changed from purple to blue and then faded gradually. (2) The weight of the microencapsulated powder decreased as the temperature increased. The weight reduction was not significant below 100 °C, but it rapidly decreased between 100 °C and 200 °C. From 200 °C to 350 °C, the rate of weight reduction slowed down, and after reaching 350 °C, the weight tended to stabilize. (3) With the increase of the mass fraction of the binder, the colour fastness of the fabric and the fabric strength also increased, while the *K/S* value of the fabric almost remained unchanged. (4) As the mass fraction of the cross-linking agent increased, the colour fastness and fabric strength improved, and the *K/S* value of the fabric decreased at room temperature. (5) With the increase in baking temperature, the colour fastness and fabric strength also increased, while the *K/S* value of the fabric almost remained unchanged.

The contribution of this article lies in the use of temperature-sensitive colour-changing microcapsules to prepare fabric, thereby achieving temperature-sensitive colour change in fabrics and providing an effective reference for diversifying textiles.

Author Contributions

Conceptualization – Zhang J and Li N; methodology – Zhang J and Li N; formal analysis – Zhang J and Li N; investigation – Zhang J; resources – Li N; writing-original draft preparation – Zhang J and Li N; writing-review and editing – Zhang J and Li N; visualization – Zhang J; supervision – Zhang J. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare no conflict of interest.

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